Dedicated to Aldous Huxley and all the courageous individuals who have come after, that dare to brave persecution in order to bring a little Light into the world.

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# PSYCHEDELIC GUIDE

## TO PREPARATION OF THE EUCHARIST

In a few of its many guises

as edited by
Robert E. Brown
& Associates
of
the Neo American Church
League for Spiritual Development
&
The Ultimate Authority of the Clear Light

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# INTRODUCTION

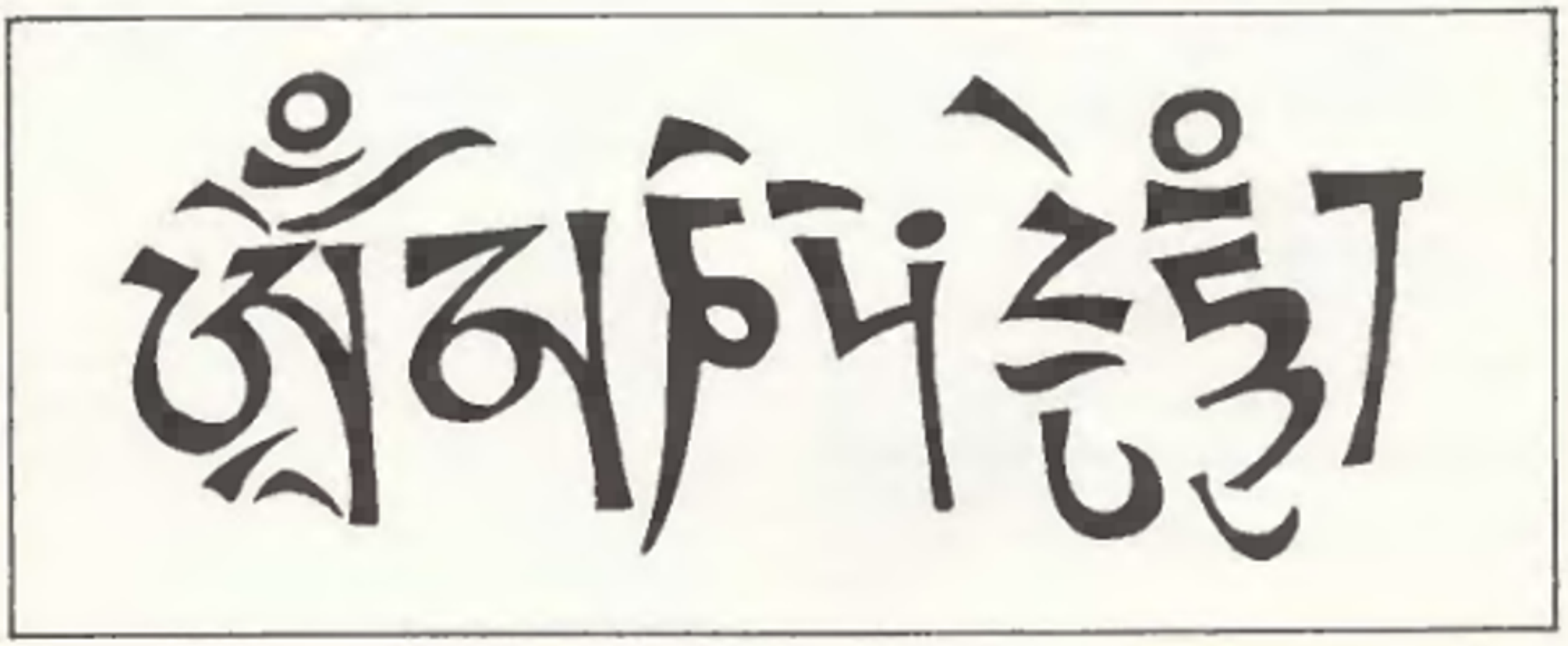
This booklet contains several processes concerning the manufacture and, to some extent, the use of various hallucinogenic chemicals. These chemicals are all restricted by federal law and possession or sale of most is definitely illegal.

Presentation of these processes is designed for persons who know how to use hallucinogens and who wish to use them for religious purposes such as the mystical Psychedelic Experience. The only other source of these drugs for religiously oriented persons is now the black market, where mixtures of variable cost and questionable quality make the hazards of using these already tricky drugs almost prohibitive.

These procedures are accurate and reliable and produce pure products of suitable quality for almost any usage requiring high standards of quality. The compounds thus produced are less likely to cause side effects due to impurities since the quality can be made to approach that of pharmaceutical house products.

Certain of the processes are given in quite technical terms with little explanation of minute details, while others are simpler and require much less knowledge of organic chemical technique to successfully carry out their directions. There is a reason for this type of layout. Some of the techniques are extremely complex and often are terribly dangerous to anyone not possessing a thorough knowledge of organic technique. Ignorant blunders could cause certain of the products to turn poisonous, and other procedures use chemicals so dangerous that flaming explosions would be the result of small mistakes. The yields in many cases are drastically reduced if proper technique and equipment are not used. Since all of these processes are to some extent chemical procedures, it is highly advisable that any person attempting to follow them with any hope of success should be proficient in organic technique and theory.

For these reasons the tricky and dangerous procedures are couched in terms designed to discourage the untrained person.



## NOTES ON THE EFFECTS OF HALLUCINOGENS FROM A PSYCHEDELIC VIEWPOINT

The brain is an immensely complex, randomly connected computer, balanced and tuned by an intricate chemical and electrical system which keeps it functional even when severely damped by alcohol or stimulated by amphetamines. However, the higher brain, the cerebral cortex, is dependent upon a constant flow of sensory input to give it a reference frame upon which it builds its various operations. Any disruption of this flow destroys the reference frame, and the cerebrum temporarily loses its ability to correlate data.

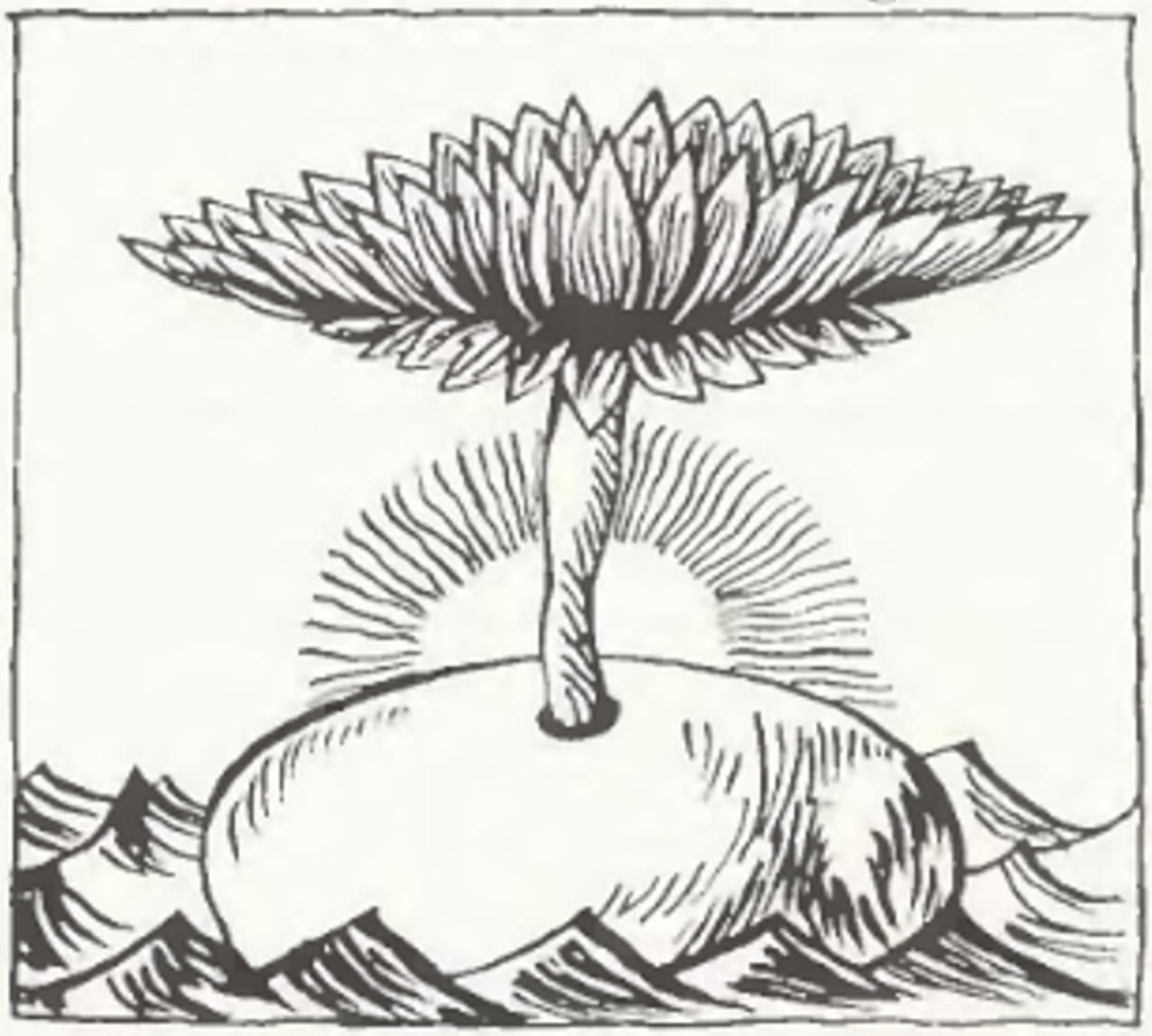
Any method which will modify or stop the feed of data to the cerebrum produces almost the same effect. The psychologist's sensory deprivation box, a physical system, removes all sources of sensory stimulation. Certain chemicals, the hallucinogens, merely disrupt the influx of sensory information by unbalancing the lower brain where the selection center for incoming data is located, giving a jumbled mass of unrecognizably crossed signals. Both methods leave the higher center with a floating reference system which it tries to maintain by developing, at random, a flow of synthetic sensory data. The mind sees these syntheses as hallucinations, and senses the loss of the reference coordinates in a feeling of disorientation with a lack of any conception of time, direction, dimension, etc.

This randomized state of the brain may most nearly be likened to that of a new-born child who has no stability, due to the absence of any coordinates at all other than inherited instincts. The main difference is that a psychedelically treated brain has what is called a mind built up in its networks, while a child is empty.

We have seen how the mind continues to try to function even when the foundations have been dissolved. The foundation, or framework, is known as the ego, the dissolution of which is consequently known as ego-loss. If the mind has carefully been coached to accept ego-loss, it will continue to progress until it finally frees itself in its complete form. The result of this final inward look is a self-awareness and enlightenment which is devastating

# the Psychedelic Guide to Preparation of the Eucharist

in a few of its many guises



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in impact and unbelievable to anyone who has never undergone the same experience.

The period of ego-loss and then the period of return of the ego are very sensitive to influences from the environment, either to the good or to the detriment of the individual's mind. The main cause of this extreme sensitivity to outside stimuli is the state of no-framework as exists in the infant. This artificial rebirth of the mind should be treated with as much care as that given the infant mind, with greatest care devoted to preliminary set and setting for each psychedelic experience. It must always be remembered how the deepest and most significant traumatic experiences come when the mind is largely unformed, as in the earliest years of life.

Other dangers in the deep psychedelic experience draw from deep conflicts and traumas which are kept hidden from the mind normally and which may appear with all the terrifying reality of the original experience. All that may have kept the mind from psychosis may have been the suppression of those unbearable experiences. Unless the mood surrounding the session, is nearly ideal coupled with complete trust in the Guru, the person could lapse into a deep psychotic fugue from which he might never recover. Very skillful handling of a session may very possibly bring those experiences out in a tranquil setting where the Guru may help the person neutralize the traumatic effect permanently.

All these considerations should be carefully looked after by individuals seeking religious enlightenment through drugs. Light doses tend to enhance moods but do not usually initiate strong hallucinogenic reactions, and are not as dangerous. Heavy doses used to bring about the psychedelic experience should never be casually played with by the novice.

quickly and efficiently absorbed into the lower intestinal tract if introduced by means of a 3% saline enema. Esthetically unpleasant as this means of introduction may be to certain people, it is one of the most efficient methods. LSD is so powerful in tiny doses that it may be taken any number of ways using foods or drink as carriers. Care should be taken to protect LSD from heat and prolonged exposure to air, especially in an aqueous solution. All crude products should be either ground very fine or vaporized into smoke.

## DOSE ADJUSTMENT AND BUILDUP

Most hallucinogenic intoxications may be adjusted by further dosage following the initial ingestion. It must be remembered, however, that the advantage of the initial shock of the drug will largely be lost, diminishing the psychedelic effect greatly. Similarly, if the dosage is repeated too often, the body remains somewhat on its guard and again the psychedelic advantage is lessened. Hallucinations will be present, but should not be confused with the full, non-game, First Bardo awakening.

Dosage adjustment is fairly simple with slow acting, long term hallucinogens. Fast-acting hallucinogens such as DMT and THC (when smoked) depend largely on the full dose being absorbed in one breath for most effective results. DMT sometimes fails entirely if taken too slowly.

Optimum repeat frequency of drugs is determined by the length of time the body takes to become off its guard to the full shock of the mind-disorienting effect. Recovery time apparently is about 15 times the length of the intoxication and is laid out for several hallucinogens in the table below. As body chemistries vary the figures will be approximate, so one should not be too concerned if recovery times or even dosage tolerances vary from person to person.

| Drug | Length of Session | Optimum Repeat Frequency |
|---|---|---|
| | | |
| DMT | 15 minutes | every four hours |
| THC (smoked) | 3 hours | daily |
| THC (eaten) | 6 hours | twice weekly |
| Psilocyn | 6 hours | twice weekly |
| LSD | 12 hours | weekly |
| Mescaline | 12 hours | weekly |
| STP | 24-36 hours | twice monthly |

## PSYCHEDELIC DOSAGE INFORMATION

All dosage information which follows purposely avoids mention of any methods which involve injection, either intravenous or intramuscular. Injection can be unsafe as far as health reasons go and does lead to easy detection by law officers who may interpret needle tracks as signs of a heroin addict. Most important, injectable drugs need the care and quality-control available only in pharmaceutical house scale production, hence are not practical in the ordinary lab.

### DOSAGE BY WEIGHT

| Drug | Light High | Experienced User | Normal | First Psychedelic | Maximum safe dose |
|---|---|---|---|---|---|
| LSD | 20-50 μg | 100-200 μg | 300 μg | 400-800 μg | 1000-3000 μg |
| THC | 1 mg | 2.5 mg | 5 mg | 10 mg | 20 mg |
| STP | 5 mg | 10 mg | 22 mg | 35 mg | 50 mg |
| DMT | 5-10 mg | 20-30 mg | 40 mg | 50-60 mg | 100 mg |
| Psilocyn | 5-10 mg | 20-30 mg | 35 mg | 40-60 mg | 150 mg |
| Mescaline | 50-200 mg | 300-500 mg | 400 mg | 600-800 mg | 1000 mg |

The very light doses only give a feeling of well-being and seldom cause much hallucination. The experienced user can achieve the psychedelic effect with less of a dose, grading off to no drug at all. A person trying to experience the full psychedelic effect for the first time needs to be disconnected much more fully, therefore the larger doses. The largest doses cause a complete break with reality that few can handle pleasantly. The slight toxic side effects are not a major factor in considering the dosage because the hallucinogenic effects are so much more overwhelming at those high doses that other factors are minimized The crude natural substances are not listed here because the amount of drug available does not follow any reliable weight percentage as the quality of these substances is not at all constant.

### METHODS

All relatively safe methods of dosage involve absorption through some of the mucous membranes. LSD, STP, and mescaline should not be vaporized by heat as they decompose on heating, but the others are more heat stable and may be taken into the lungs as a vapor, usually through some form of smoking. The mucous membranes of the digestive tract take in all but DMT efficiently, THC and its cruder forms, marijuana and hashish are fat soluble and enter most efficiently if dissolved in butter or cooking oil, which acts as a carrier and enters via the fat absorption process. DMT and THC excepted, all the hallucinogens are

## ADJUSTING WITH OTHER DRUGS

Other drugs should be used with great caution. Sea sickness remedies like Dramamine will help combat nausea. Librium calms nervous individuals in the pre-session period. If severe trauma develops and none of the verbal methods work, then chlorpromazine or other phenothiazine tranquilizers may be used to stop the hallucinatory effects. This should not be used with STP as the effects are sometimes heightened. Niacinamide (3 g) and ascorbic acid (3 g) combined will abort a bad LSD trip in about an hour. Monosodium glutamate has a slight effect in repairing mental tone after a session.

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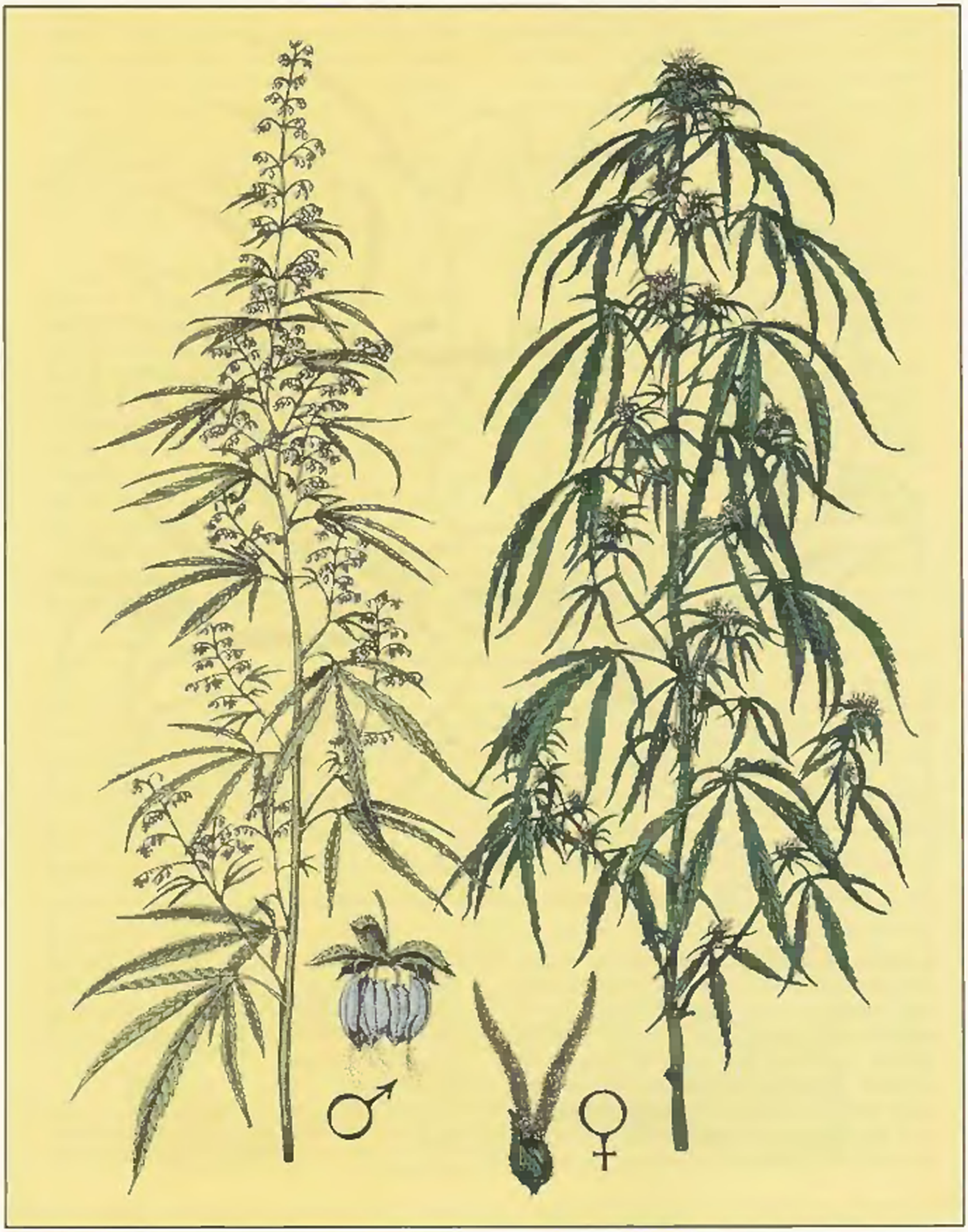
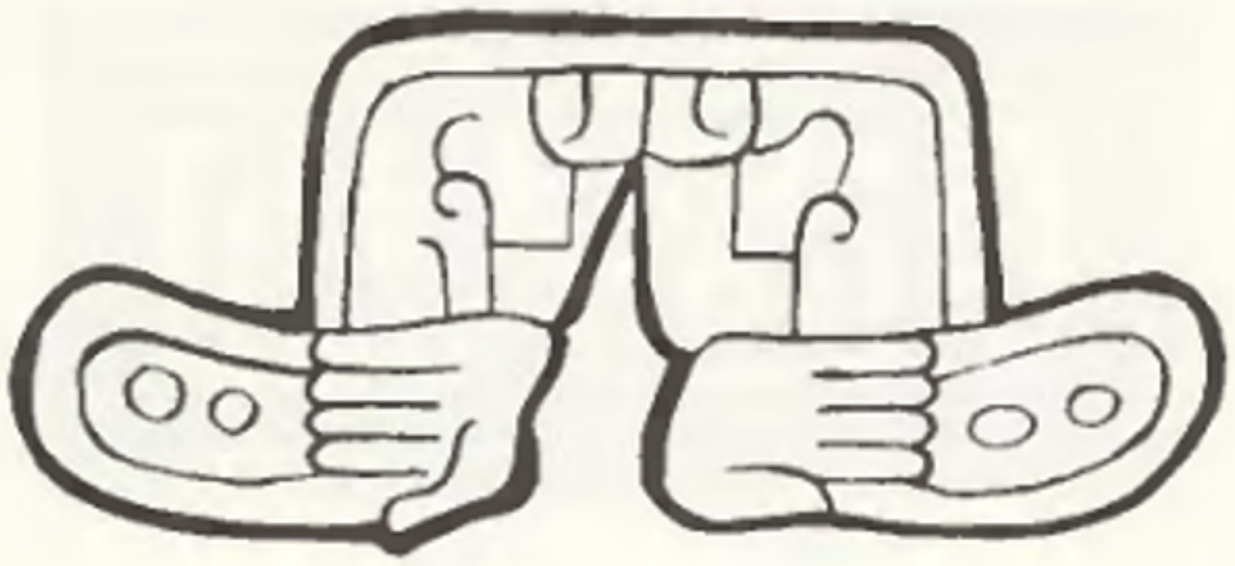




## CULTIVATION OF MARIJUANA

Marijuana grows in almost any climate, but prefers a hot period at the end of the growing season. It grows best in loose, fertile soil, but will survive in almost any basic soil, preferably with adequate fertility and moisture which are easily provided. The tap root goes deep, so the soil should have a depth of at least three feet above bedrock.

Select only the best seed. Wild marijuana is usually of an inferior strain, so select seed from a good Indian strain or from the best Acapulco Gold. Some seeds from cured marijuana may be nonviable, so each batch must be tested by germination of a sample in a rolled, damp towel set in a warm place for a few days.

When danger of frost is over, plant the seeds ½ inch deep and three inches apart in well mulched, well turned, slightly basic, fertile soil. The seeds will be up in about three days, at which time they must be watched so that slugs, isopods and cutworms do not eat them. After the seedlings are two weeks old, little care is needed to protect them, as the hemp plant is extremely hardy. The seeds may be started in individual containers indoors and set out when they have a good start.

If the plants begin to show excessive height, they may be topped by pinching the apical bud, making them grow bushy rather than tall. Late in the season the plants will begin to show differentiation into male and female. The female plants, which have the most tetrahydrocannabinol, are sturdy, dark green bushes several feet tall with leafy bracts all over the upper stems hiding the female flowers and seeds. The male plants become somewhat tall and spindly and are lighter green, with the male greenish-yellow flowers dangling in the breeze. Male plants, to derive any good from them, must be taken at first sign of their flowers. When the female flowers have bloomed and before the seeds have set, that portion should be harvested. Female flowers are hard to see and must be examined closely to follow their progress.

Pick the upper leaves, bracts, and flowers from the female plants. Dry in a warm well-ventilated place, preferably not in direct sunlight. When thoroughly dried, the material is cleaned and cut for smoking. To keep any length of time, seal in an airtight container with 5% by weight of 95% ethanol (Everclear).

The Federal Narcotics brigade doesn't like people to grow marijuana in the United States and will arrest anyone who allows the plant to grow on his property. Many times meter readers are instructed to look for marijuana and report violators to the Bureau. It is still possible to grow marijuana in Mexico, although most areas have sporadically enforced laws regarding it.

## HASHISH
## AND BHANG

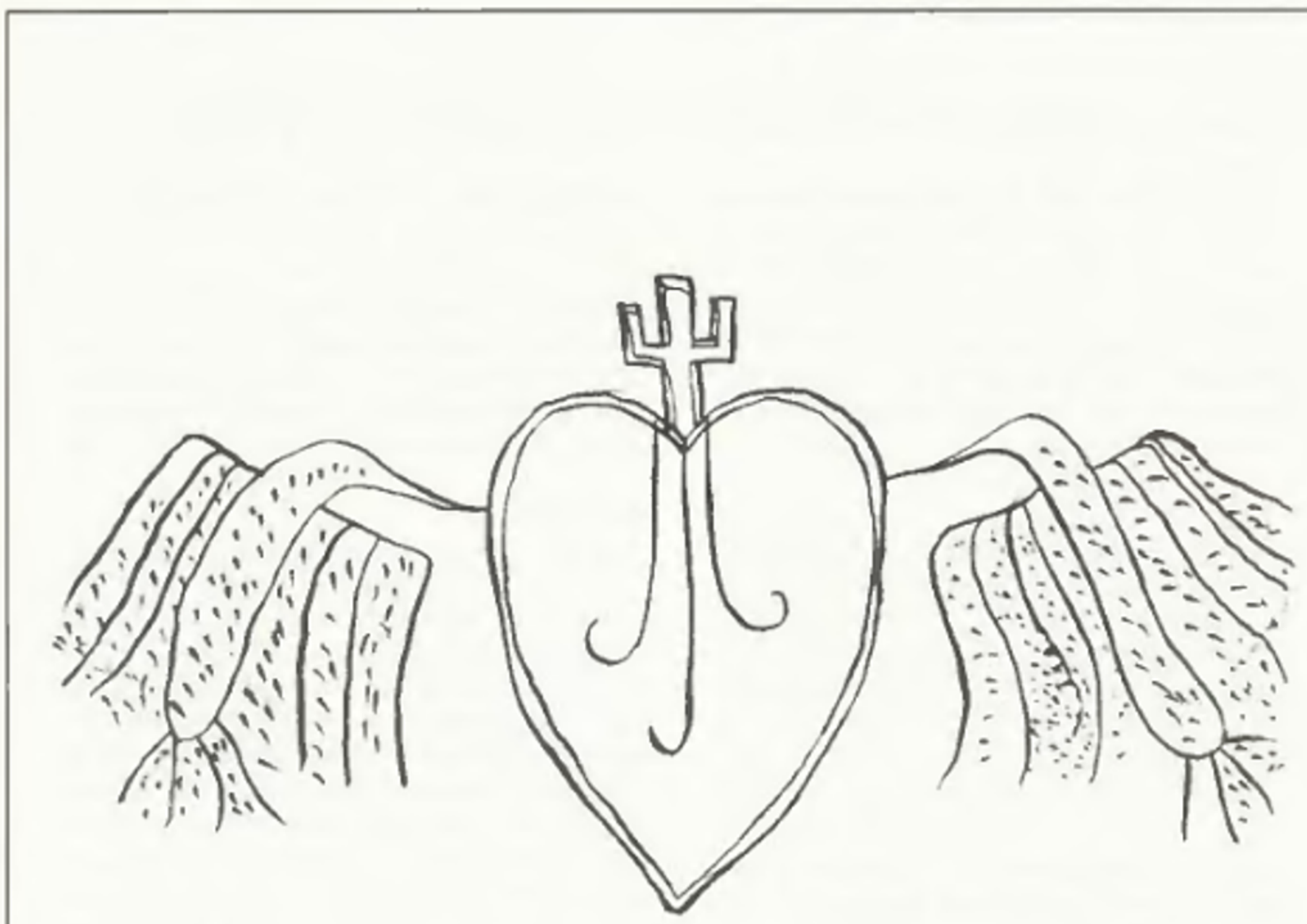
Bhang is the dried, crude, water extract of the hemp plant, *Cannabis sativa*. This product is commonly prepared in eastern countries from both low and high grade plants. Plentiful raw material is available in the innumerable hemp fields of the central United States.

Indian processors gather the whole plants at time of flowering, and with no further treatment the plant material is macerated in a crusher-roller similar to that used in sugar cane processing. The crushed hemp is steeped in boiling water with adequate stirring so as to leach the suspendable solids from the fibrous and woody portions. The murky, greenish solution is simmered slowly to reduce the volume, and the syrupy residue is dried in the sun or in a drying oven. The resulting mass is rolled into lumps or molded into blocks for sale. This product is made to be dissolved in a drink but it is sold for hashish in the United States. Hashish is made by rubbing the sticky green plants, then rubbing the hands to roll up the sticky resin. This is molded into blocks.

A more practical process in this country consists of drying the plants and stripping the leaves and flowers from the large stems. This leafy material is packed into a large jug or barrel and soaked with either alcohol, acetone or a purified petroleum solvent. If many seeds are present the petroleum solvent should not be used. A siphon fitted with a strainer put in the container before filling makes removal of the extract much easier. The solvent is evaporated and the hashish shaped into blocks. A second extract should be made on each batch, the solvent from that going into fresh plant material as the first extract solution. A third extract may be made, that solvent being saved as the second extract solution for another batch. The soaking time should be at least overnight for best results.

For use, the Bhang or hashish should be physically modified since the solid block form is an inefficient form either to eat or to smoke. The block hashish may be shaved with a sharp knife, or, if a large quantity of Bhang is involved, the material may be cut thin with a hot knife, dried in a 100°C oven, and pulverized in a blender. Powdered Bhang is easily blended into smoking mixtures, or an alcoholic tincture of hashish or bhang may be made by taking up the soluble portions into four times the weight of warm 95% ethyl alcohol.

To activate hashish or bhang (or marijuana) and to prepare it for use in cooking, simply heat the Cannabis preparation with the fat desired in any recipe, plus a little water. Then simmer the mixture carefully over low heat for several hours, being careful not to scorch the materials. The oily portion will have turned an olive green and will have most of the active principles dissolved in it. This method triples the efficiency of hashish used for eating purposes.



## MARIJUANA RED OIL EXTRACTION

The female flowers and leafy tops are collected before the seeds have set. Be sure to dry it in a place with adequate ventilation to prevent molding. No curing is necessary in preparing marijuana, as the strength of the preparation is entirely dependent on the variety grown. In this process any grade, even wild material, may be used since it is to be concentrated and a poor grade merely gives less yield per pound.

The dried, ground material is extracted repeatedly using a hydrocarbon solvent preferably, although alcohol is sometimes used. A low boiling solvent is most convenient since it is removed from the extract most easily. The most economical solvent is one of the lighter fluids or a gasoline type, highly volatile cleaning fluid. The extract is filtered and concentrated by evaporation or careful distillation, taking great care to avoid any flame in the vicinity.

The gummy deposit obtained is quite potent, but further purification is possible. Steam distillation removes the odorous terpenes. The dried resin may then be distilled at $5 \times 10^{-3}$ mm Hg pressure. Collect the fraction BP 70-160° and stop when the residue begins to decompose. The distillate is marijuana red oil, very active physiologically.

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## SYNTHESIS OF TETRAHYDROCANNABINOL TYPE COMPOUNDS

The synthesis of tetrahydrocannabinol is usually somewhat tricky if intermediates are not available. The essential portions of the molecule which must be obtained for the final condensation are an isoprene series essential oil, such as pulegone or citral, and a5-alkyl resorcinol. The latter compound is least common and most likely to be watched.

**PROCEDURE I** – One of the most direct syntheses is a one step procedure, stirring 18g olivetol with 15 g citral in 100 ml benzene at 5-10 degrees, containing 10 ml of boron trifluoride etherate. The reaction should proceed one hour, then the mixture is extracted with dilute alkali (1% sodium hydroxide) to remove the unreacted olivetol. To purify, chromatograph on a fluorisil column with hexane to remove the first isomer which is inactive, but not poisonous. Subsequent elution with a 95-5 solution of hexane-ether will bring off two more isomers together, one of which is active. The solvent solution, when evaporated, leaves a resinous residue very similar, if not identical, to the active components in marijuana in very concentrated form.

**PROCEDURE II** – This method uses pulegone instead of citral, and butyl lithium as the means of effecting the condensation.

To butyl lithium prepared from 2.5 g lithium and 20.5 g of anhydrous n-butyl chloride, add 42 g of dry methoxylated olivetol, carrying out the reaction under nitrogen gas. After shaking for three hours, 25 g (26.8 ml) of dry pulegone in 50 ml anhydrous ether is added and fifteen minutes later the mixture is cautiously decomposed with ice water. Extract with ether. Distill at 1 mm after the ether and water are taken off. The methyl groups may be removed by warming an alcoholic solution with hydriodic acid.

## SYNTHESIS OF 5-ALKYL RESORCINOLS

Any of the 5-alkyl resorcinols may be substituted for olivetol in these methods. In the following procedures, along with olivetol, is a complex synthesis of a 5-alkyl resorcinol which will give an end product which is 50 times as active as the natural product.

### OLIVETOL

#### Step I – 3, 5- Dinitro Butyl Phenone

Prepare the grignard reagent from 50 g 1-chloro butane by refluxing with 20 g magnesium turnings in 500 ml dry ether solution. Take precautions concerning ether fumes escaping and air entering the flask by fastening a long tube from the reflux condenser to the outside air. A crystal of iodine may be necessary to initiate the reaction. When the reaction is complete, add 92 g anhydrous cadmium chloride and continue the reflux another 15 to 20 minutes. The ether is removed using vacuum and to the residual Cd complex is added 500 ml benzene, taking care to keep excess oxygen out since the compound may spontaneously take fire in air. To this mixture is added 115 g (0.5 Mole) of 3, 5-dinitro benzoyl chloride. Reflux for 15 minutes and distill off the benzene. Recover the ketone using distillation under reduced pressure, taking the largest fraction.

#### STEP II – 1(3, 5-Dihydroxy) Phenyl Pentane

Prepare the Clemmenson zinc by dropping molten zinc in water. Immerse 160 g of these granules in a 5% mercuric chloride solution for two hours. Decant and rinse with water. Place the amalgam and 62 g (0.25 Mole) of 3, 5-dinitro butyl phenone in a large flask and add a mixture of 400 ml water and 200 ml con HCl. Attach a reflux condenser and gently

heat the mixture until a vigorous reaction starts. When the reaction has slowed, continue the reflux for three more hours, adding 15 ml of con HCl every hour. Remove the unspent amalgam, cool and add 50 ml con sulfuric acid and 50 g of sodium nitrite keeping the temperature near 15 degrees for the first half hour. Allow the temperature to rise to 95 degrees and heat at that temperature for 2 hours. Extract the olivetol with three benzene washes, remove the benzene and purify by distillation and under reduced pressure. BP 180° at 6 mm.

## A HIGHLY ACTIVE EXOTIC 5-ALKYL RESORCINOL

### STEP I – 3, 5-Dihydroxy Benzoic Acid

To a suspension of 130 g powdered iron in 500 ml water containing 40 ml con HCl is added 100 g 3, 5 dinitro benzoic acid and the mixture is refluxed for two hours. The acidic crude aniline derivative is filtered and the filtrate acidified with 100 ml con sulfuric acid and 75 g sodium nitrite is introduced. The reaction should be kept near 5-10 degrees for one half hour when the solution is slowly brought to 95 degrees and held at that temperature for one hour to thoroughly decompose the diazonium salt. Extract the product with several washings of ether. Remove the ether and recrystallize the 3, 5 dihydroxy benzoic acid from hot alcohol.

### STEP II – 3, 5-Dimethoxy Benzoic Acid

2 g of sodium sulfite (or a nitrogen flush) is added to a 100 ml RB flask containing 500 ml cold water with 60 g (82 ml 50% sol) of sodium hydroxide and the flask is lightly stoppered and shaken for ten minutes. To this solution is rapidly added 77 g (0.5 Mole) of 3, 5 dihydroxy benzoic acid. When this solution is dissolved, cool the flask in a cold water bath. 63 g (47 ml) of dimethyl sulfate is then added and the flask stirred for 20 minutes. Care must be taken to keep the temperature low. 63 g (47 ml) more dimethyl sulfate may then be added, allowing the temperature to rise to 45 degrees. Reflux this mixture for one hour, at which time 10 ml of 50% sodium hydroxide made up to 25 ml is added and reflux is continued another hour to saponify any ester that may be formed. Cool the solution and acidify with con HCl. Vacuum filter the ice-cold solution and dry the precipitate in warm air.

### STEP III – 3, 5-Dimethoxy Benzoyl Chloride

To 500 g of 3, 5 dimethoxy benzoic acid, add 310 ml freshly distilled (pure) thionyl chloride and heat the mixture two hours in a water bath. The mixture is distilled under reduced pressure using all glass joints. Yield is 510 g, boiling at 150 degrees (18 mm).

### STEP IV – 3, 5-Dimethoxyphenyl 2 methyl heptanone

This step is a Grignard reaction and the apparatus should have a long vent tube from the reflux condenser to vent ether fumes and prevent air from entering the reaction. A couple of iodine crystals help to initiate a balky reaction.

134 g (1 Mole) of dry 2 chloro heptane is refluxed with 24 g of Mg turnings in 500 ml anhydrous ether in a 1000 ml RB flask until the reaction is complete. Cool this solution to -65 degrees with a dry ice acetone bath and add 25 g anhydrous ferric chloride. To this cold mixture add 107 g (0.5 Mole) of the 3,5 dimethoxy benzoyl chloride from step III. Stir for several hours at -65 degrees and at the end of this period add finely ground ice, allowing the temperature to rise to 25 degrees. Remove the ether and recover the ketone using distillation under reduced pressure, taking the largest fraction. Any tertiary alcohol produced may be used as is or may be reduced to the alkene by passing over pure activated alumina at 350-450 degrees, with

subsequent reduction with sodium and absolute alcohol. This derivative, while not as potent as the main product, is at least as active as olivetol.

### STEP V – 1(3,5-Dimethoxyphenyl) 1, 2 Dimethyl 1 Heptene

Dry the ketone over anhydrous sodium sulfate. Prepare a methyl grignard reagent by reacting 95 g (54.7 ml or one Mole) of methyl bromide with 20 g Mg turnings in 500ml anhydrous ether at a low temperature (methyl bromide boils at 3.56 degrees). 150 g (0.5 Mole) of the ketone from step IV is combined with this methyl grignard reagent and the solution is refluxed for 15 minutes, whereupon 100 ml of water containing 1 ml of con sulfuric acid is added. The 1(3, 5 dimethoxyphenyl) 1, 2 dimethyl heptene is distilled after the ether and water has all passed off. BP about 180 degrees.

### STEP VI – 1(3, 5- Dimethoxyphenyl) 1, 2 Dimethyl Heptane

Dissolve the dry heptene derivitive in absolute ethanol and reduce to the 5-alkyl methoxylated resorcinol with sodium metal. For the tetrahydrocannabinol procedure I, reflux the product with hydriodic acid to remove the methoxy groups.


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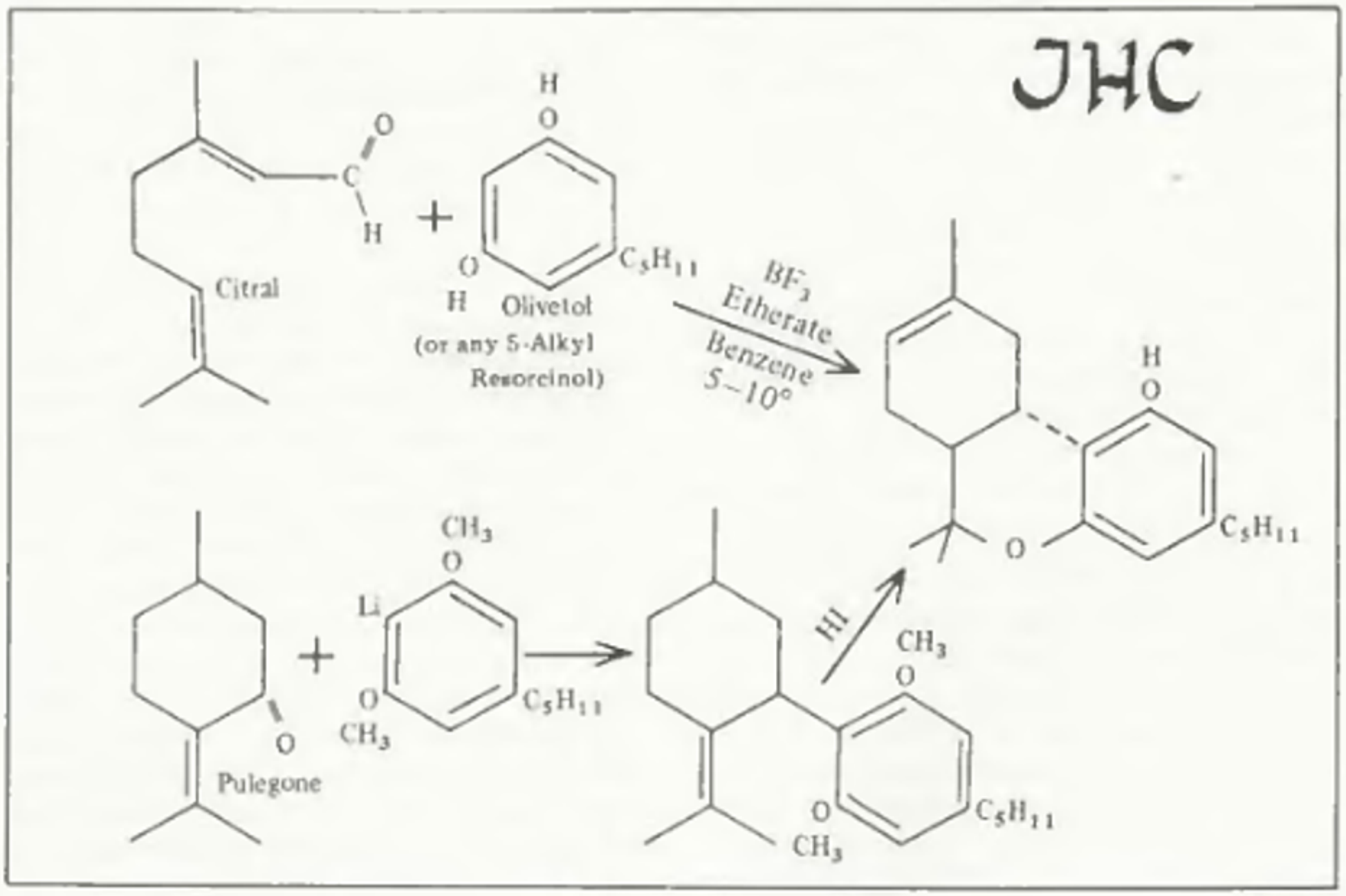
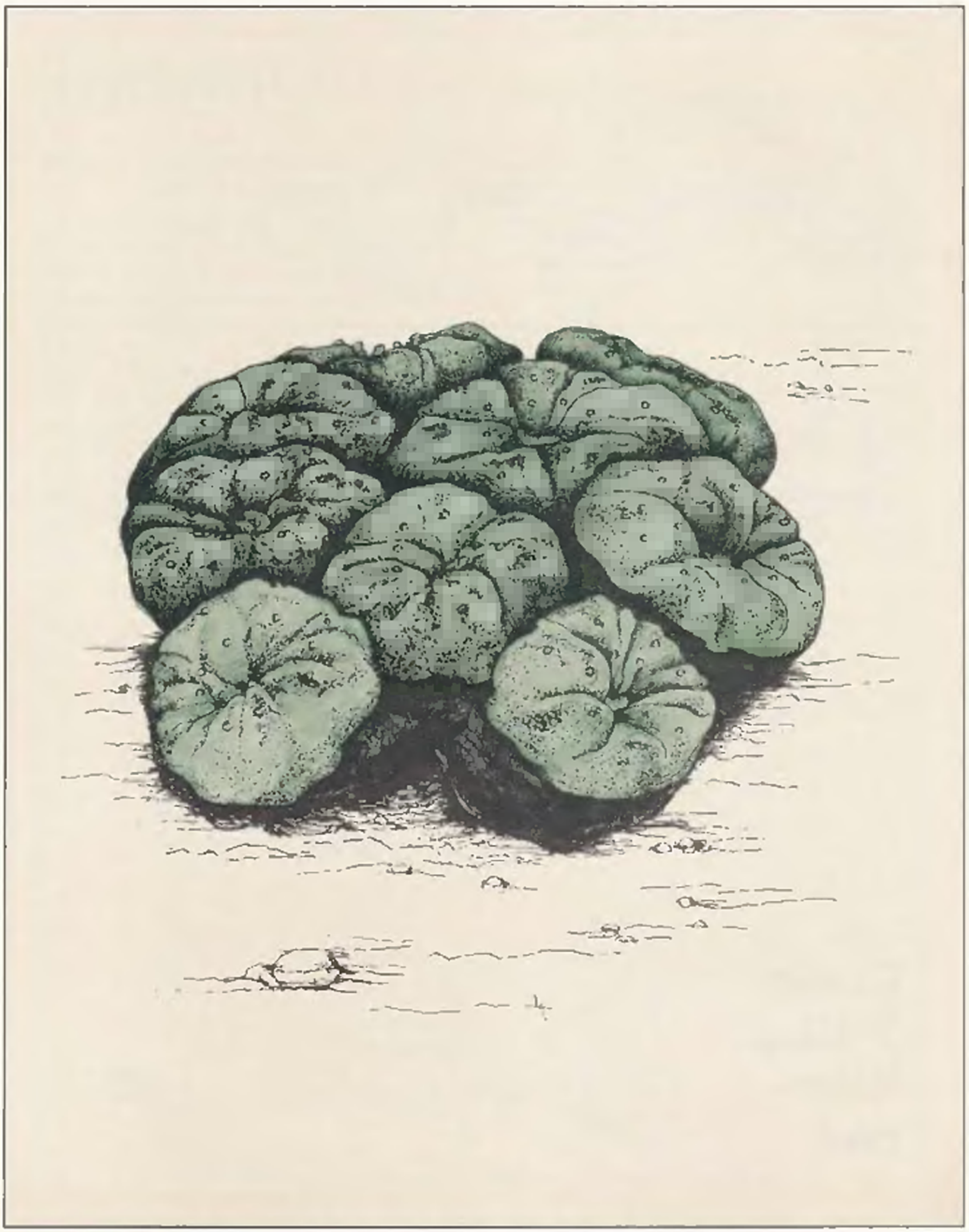
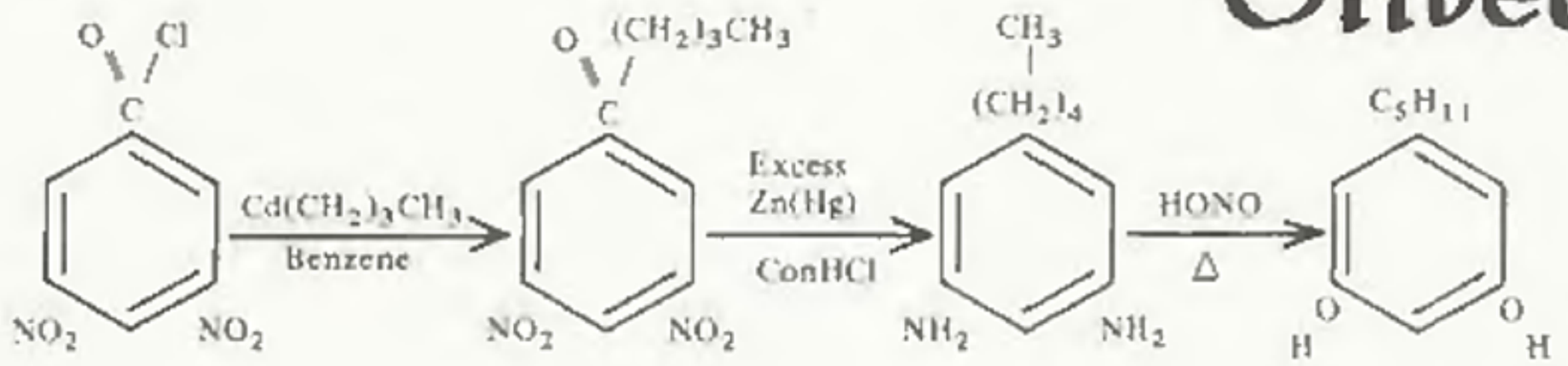
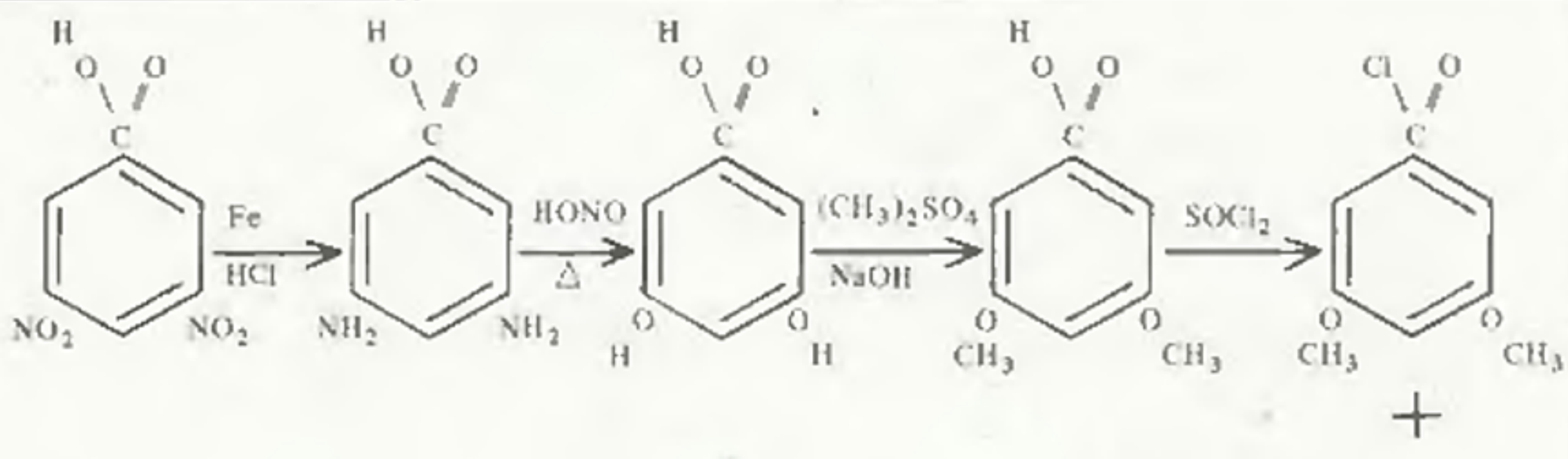
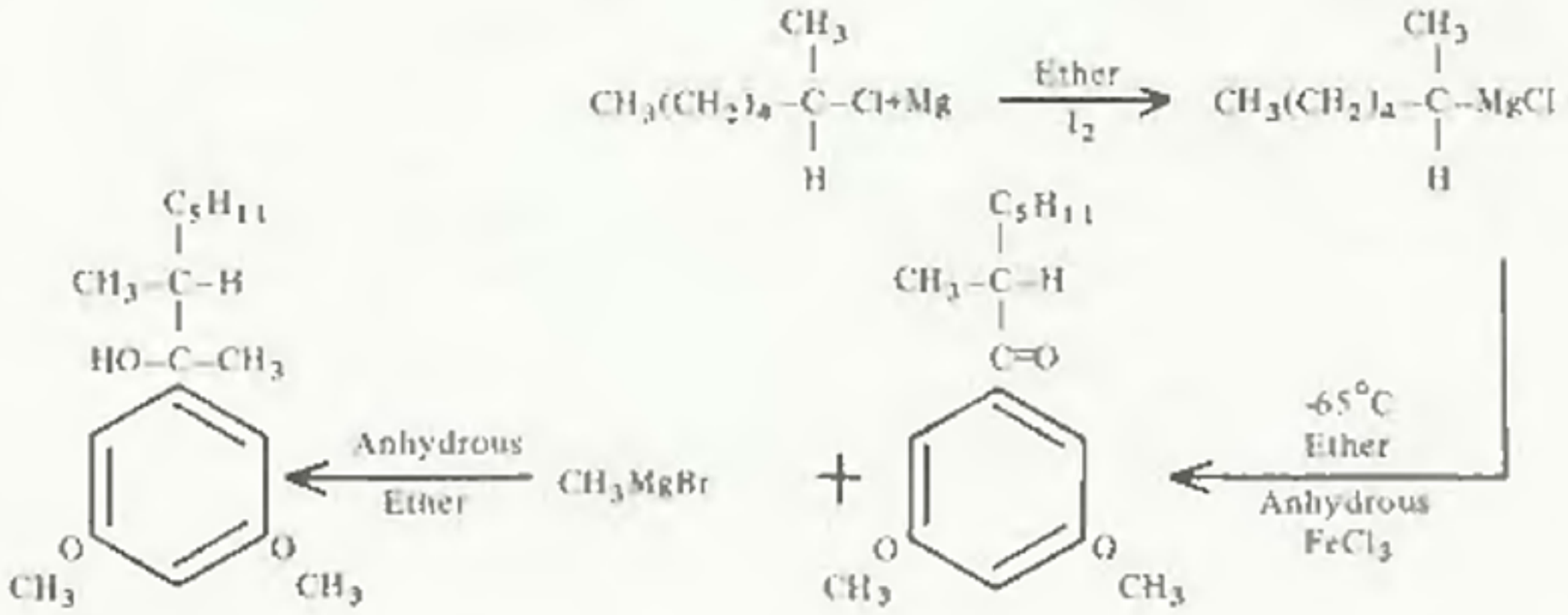


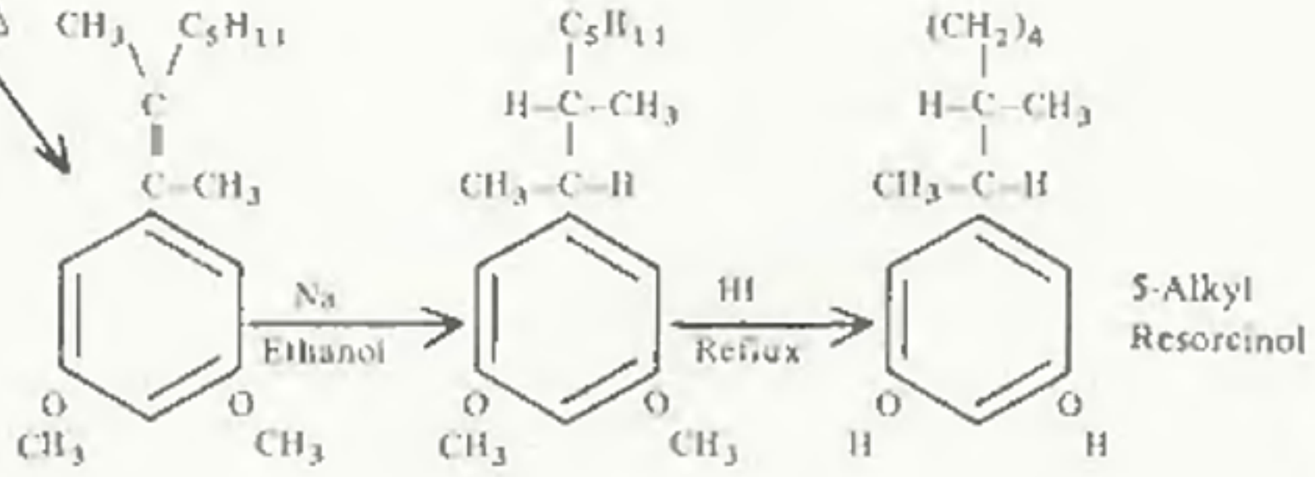


# Olivetol




## Exotic 5-Alkyl Resorcinol

## PEYOTE CACTUS EXTRACTION

The mature plants are dug or cut preferably at the end of the dry season before the rains begin. The plants apparently use the nitrogenous alkaloids in growth when adequate rain is available. Consequently the end of a long dry spell is best, so much so that yields drop below 50% if plants are harvested in a wet time. Cacti thrive on fertile soils rich in nitrogen and plants from good soils consequently produce extra alkaloids. Old large plants also give the best yield.

The plant is shaped like an ice-cream cone; the cone part being the root and the ice-cream being the green alkaloid-laden top of the plant. The root contains mostly carbohydrates and cellulose so that the very small amount of mescaline obtained therefrom is almost impossible to extract. Since over 99% of the alkaloids are contained in the green top and since the cellulose bulk reduces the extraction yield, the root may be removed with little reduction in, and probably, an increase in yield. If the roots are left in the ground, more plants will grow.

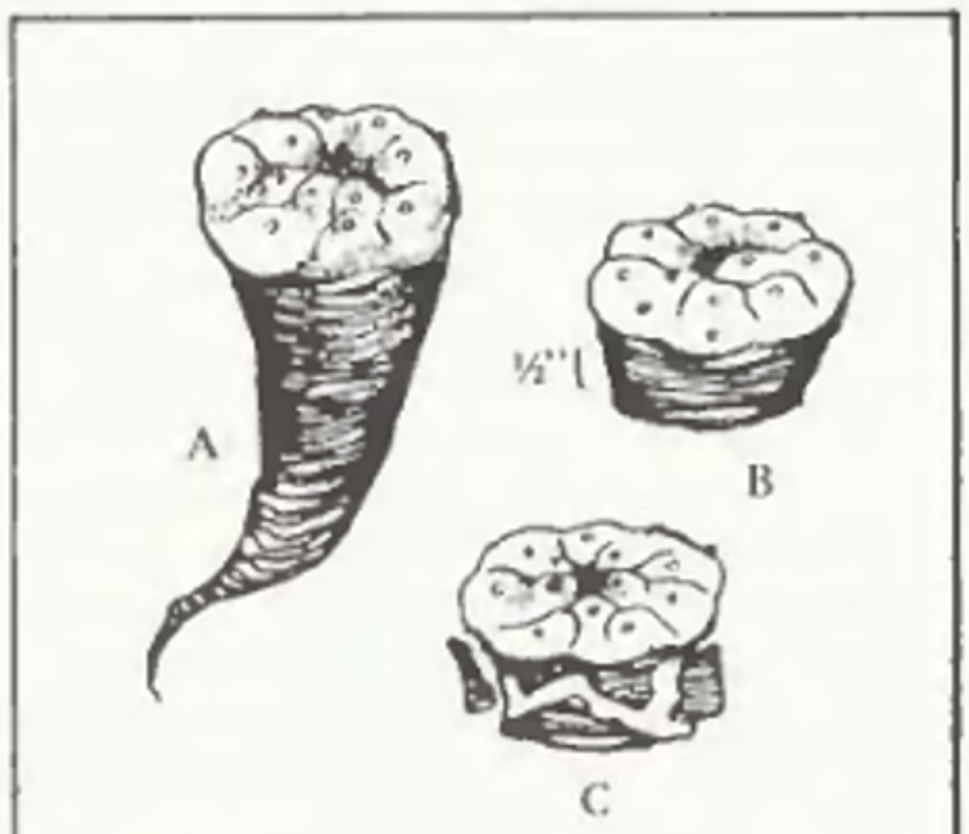



FIGURE 1

To prepare the plant, make the slice (see Figure 1) ½ inch below the line which separates the green top from the root bark. The sliced off buds still have that ½ inch of upper root covered with useless cellulose and dirt, which is removed by pinching off chunks of the hard bark from the tough, flaccid plant body. Rinse the plants briefly to remove loose dirt and still avoid leaching the plant material. Do not bother with the cottony tufts; they are a nuisance, but not enough to warrant removal. If plants are kept any length of time, treat them by sprinkling with naphthalene flakes. Remove this preservative thoroughly if the plants are to be used fresh. This basic treatment is the initial step to all other preparation processes.

If the cacti are to be stored as crude plant material, they must be dried. Even then they will retain potency only up to two years. The best procedure for drying the buds is to crush them between wood and dry them in the sun or in an oven at 120-170° F. Sun drying works best if the climate is dry, but a kitchen oven turned down low with the door open will work. The dried buds may be stored this way or may be ground to flour. Sometimes the cottony tufts are sifted out and the powder is encapsulated. However, one would need between 18 and 25 #000 capsules full to get 400 mg equivalent of mescaline. An alternative would be to make up 8 g of the powder into a solution and take it as an enema. 20 g of powder equal 1 g of mescaline.

The cactus is often taken in its raw state and choked down, but due to bad and bitter flavor, coupled with the emetic action of the drug, other methods are sought. The simplest of these involves making a pulp of the green plant in a blender and diluting with lime jello, tomato juice or other masking agents. Here again the best solution is to use an enema.

acids in the crude extract; and in turn, is extracted from the solvent by conversion to the salt of an acid.

To release mescaline as a free base, caustic soda is used since mescaline is exceeded in basicity by few bases less strong than NaOH. The caustic is made carbonate free by dissolving it in water to make a 50% solution (6 lbs./gal.). The carbonate settles out in 24 hours and pure NaOH solution is decanted. Keep away from air as it will pick up $CO_2$ and fix it as carbonate.

To every four volumes of the cooled extract concentrate add one volume of caustic solution. Mix in a large jug and immediately add at least two volumes of benzene. Ventilate the area well when using benzene due to the extremely poisonous nature of benzene fumes. Close the mouth of the jug tightly and invert smoothly 50 times or more to mix solvent with the extract liquor. Do not shake, since this produces a thick emulsion which is almost impossible to break. If an emulsion does form, let stand an hour and stir very slowly to help break bubbles. If necessary, add more benzene. Do not allow the mixture to stand more than four hours if possible, since caustic will hydrolyze mescaline in time. When part of the benzene has layered, draw most of it off by means of a pressure started siphon (see Figure 2). Collect it in a bottle and allow any residue

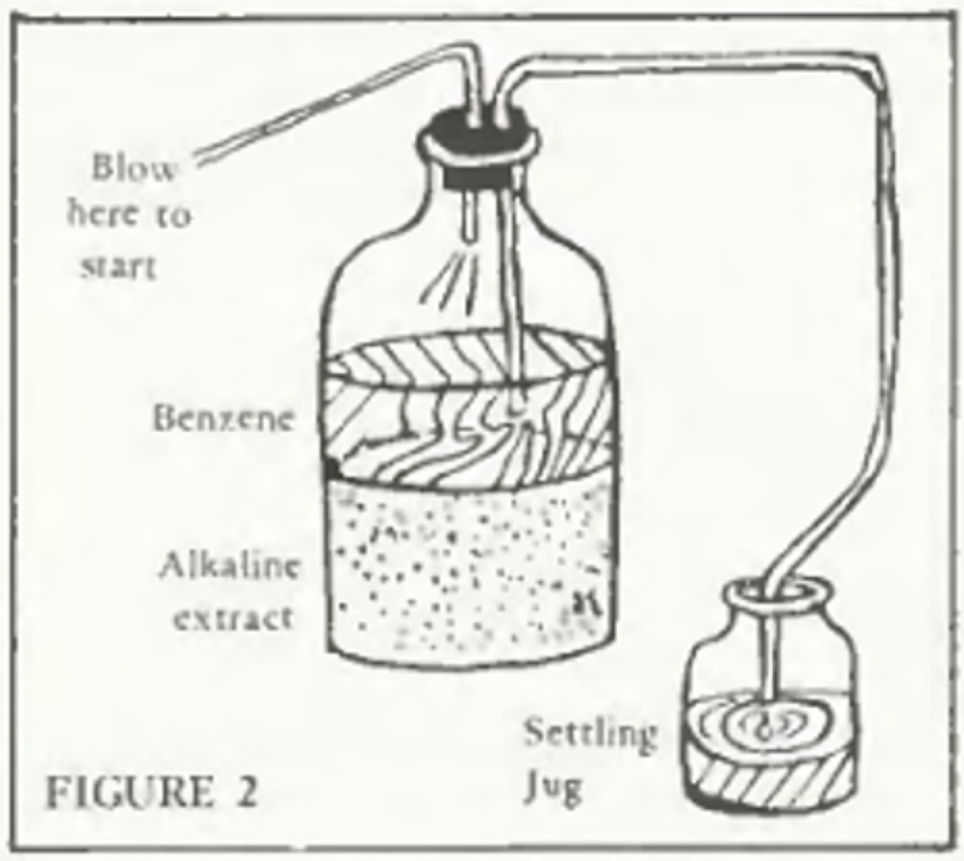


FIGURE 2

to settle. Pour the benzene from the settling jug into a clean jug, add a few drops of 10% $H_2SO_4$ (2N) and shake well. Test the benzene solution with pH paper to see if it is still basic. Keep titrating the benzene in this manner with acid until the benzene begins to turn neutral. This titration is best carried out using some kind of mechanical stirrer such as a magnetic stirrer. Over-acidity means bicarbonate must be used during recrystallization, which introduces more contaminants in the form of mineral salts which are a nuisance. A small amount of mescaline left in the benzene, making the solution slightly basic will not hurt at all since the next step is to return the used benzene to the crude basic extract solution for further extraction. No new caustic is added, and mixing is done carefully as before. The first extract yields about 80% of the total alkaloids; the second, 16%; the third, 3.2%; and the fourth, less than 1%. The crude extract is exhausted and may be discarded after four extractions, but the used benzene may be distilled to get rid of the waxes and gums.

The layer under the benzene in the acid extraction contains mushy, impure alkaloids, benzene and water. Warm this gently, using a flameless heater and good ventilation. Pour off most of the separated benzene and evaporate the last bit off over a water bath. When all the crystals are dissolved, wrap the vessel in a towel to insulate it. When this slow cooling has taken the temperature to ambient, place the insulation and all into a refrigerator and continue cooling down to 0°C. If crystals have formed, the masses are mushed up to make filtering easier. The Buchner vacuum filter funnel, almost a necessity to the procedure, is prepared by wetting the filter paper and sucking it into place with the vacuum aspirator system (see Figure 3). Filter the crystals and wash them with ice-water, then acetone. Remove the mother liquor to a warm dry place where it may evaporate to a low volume. Redissolve the dry crystals in boiling water and

At best these dosing methods are an ordeal for most people, and many methods of concentration other than drying (from the Indians) have been developed. The laboratory procedure has always begun with the dried ground plants. These plants are extracted with warm alcohol containing a small portion of $NH_4OH$ (0.5%). The liquid is then strained off, the solvent removed by distillation, and the residue treated for separation into component alkaloids. For use, the tarry residue may be rolled in cornstarch into a rope and encapsulated, or sent to the next stage of concentration (see following discussion).

The cheapest and quickest method of treating the cactus is water extraction of the fresh or dried plant material by any number of techniques. The main objective is to disrupt the cell walls, solubilize the alkaloids, and remove them in a water solution from the cellulose residue as quickly and efficiently as possible. All spills are sopped up and returned for continued treatment in the current step. The alkaloids are susceptible to breakdown by molds, bacteria, heat, and acid or base hydrolysis. Therefore the following techniques: (select the best one)

1. The raw plant tops are reduced to a pulp in a Waring blender, acidified to 0.1% acid with citric acid, heated to boiling, strained hot through parachute material and squeezed dry. Re-steeping in acidified boiling water and re-pressing will remove almost all traces of alkaloid. Discard the pulp.

2. Freeze the buds to rupture the cells, thaw, acidify as previously noted, heat to boiling, hot-press whole by placing in a cloth sack and using some kind of machine like a cider press for the pressure needed to squeeze all the liquid out. A ricer or fruit squeezer works for small amounts.

3. Pressure cook the whole plants, acidified as before, at 20 lbs. for 15 minutes to disrupt the tissues with little alkaloid loss. Hot press through cloth as in #2. Re-boiling 15 minutes in a small amount of acidified water two or three times removes all possible alkaloids for any of these three methods. There is no need to cut up the plants in either #2 or #3 since the treatment leaves the plant material like a sponge. Do not overcook. Discard the spent buds.

These procedures net a bitter liquid. This is often drunk straight, mixed, gelled or taken as an enema. The bulky pulp has been removed but the liquid volume is still a problem. Gentle boiling or simmering will reduce the volume 90% or more. A resinous scum may form. Skim, wash the scum by mixing with hot water, strain and save the washings. Using a fan and an electric stirrer will speed evaporation while keeping the temperature lower. The concentrated liquid will keep a few days with 1% benzene shaken in as a preservative. This is used only in further extraction. However, some wish to reduce it to tar and use it or store it in the form of a tar. In this case do not add a preservative, but evaporate it to tar immediately. Six or seven #00 capsules contain almost 400 mg of mescaline. A coating of shellac on rolled pills of the dryed tar (or on capsules) makes a good enteric coating to prevent loss through vomiting. The tar will keep for several years.

The thick concentrated liquid must be acidified prior to extraction, since carbonate ions interfere with solubility of free mescaline. Experiments show that the amine-carbonate to free-amine equilibrium in NaOH solution is high enough to interfere with the efficiency of extraction. Acidification of the plant with 2 oz. citric acid per 5 gal. lot before cooking will free most of the alkaloids and destroy all carbonate by the time the solution is concentrated. Benzene (one oz./gal) may be used as a preservative only if the liquid is to be used for extraction, because of its extreme toxicity.

For extraction, the mescaline must be released as a free base; extracted with a solvent to release it from the plant sugars and amino

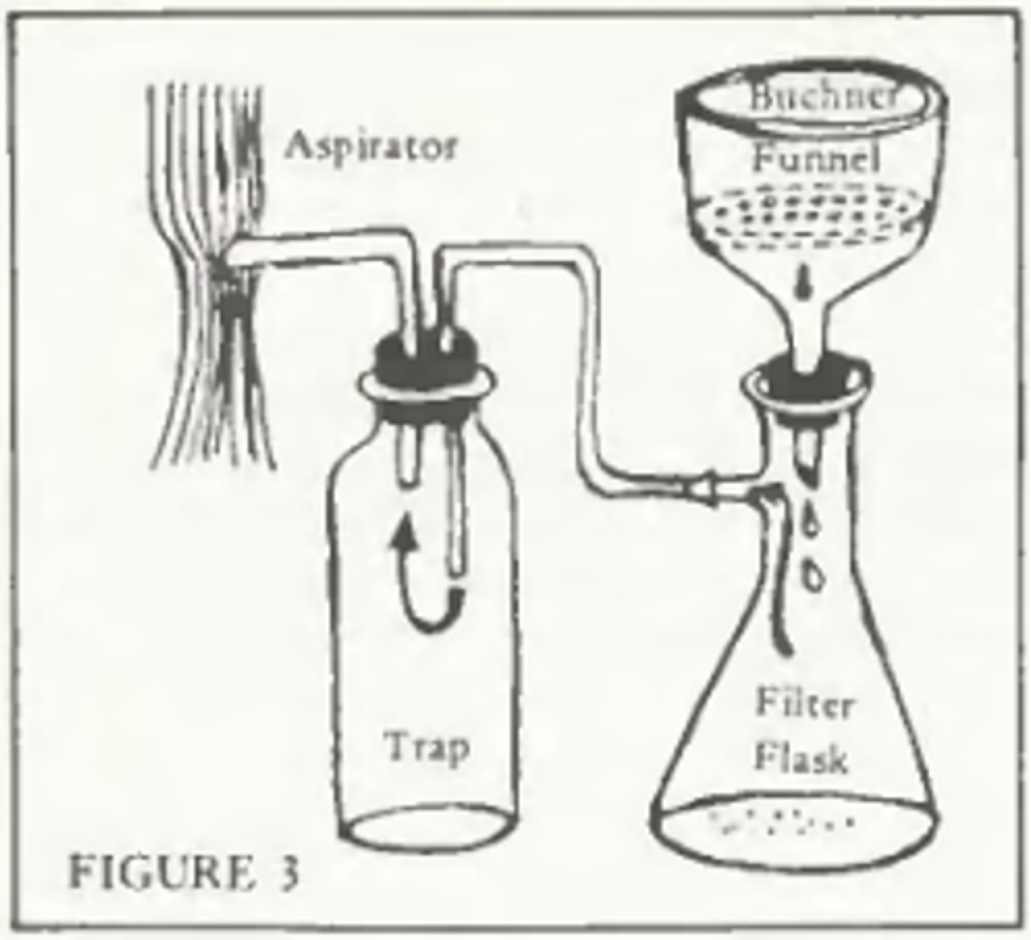


FIGURE 3

add one tsp. activated charcoal per 100 ml of solution. Prepare the filter same as above, but layer some acid-washed asbestos pulp over the paper to help seal it. Filter the solution very quickly and very hot. Keep vacuum on as long as there is charcoal liquid in the funnel to prevent leakage into the filtrate. Wash the filter with a small amount of boiling water. Don't worry about diluting the filtrate too much; if the solution is too concentrated it will not crystallize right and possibly the filter may clog up. Heat the vessel with the solution in a double boiler to re-dissolve any crystals formed prematurely. Crystallize as before. If the solution was treated properly, white needles will grow in a clear to light yellow mother liquor. The slower the crystallization, the larger the needles. Crush the crystal masses and filter, washing the crystals with ice-water and then acetone. Dry the product briefly in a gentle, warm oven (130°F) and store in tightly closed bottles.

To get a better yield, one must go back and pick up any loose ends where extra mescaline might be. The first thing to do is evaporate the last mother liquor taken from the pure crystals, and get a couple more crops out in this manner. Each time the water solution is cooled to 0°C, 3% mescaline is left. Finally a solution remains which is slightly brown or else just doesn't produce good crystals. This is evaporated to dryness and saved for later.

By this time the brown liquid from the very first crystallization should be much reduced in volume. Heat this black liquid to dissolve solids and add acetone cautiously with stirring. Use 10 times the volume in acetone. Cool, filter and wash with acetone. Combine this with the slightly impure solid above, dissolve in boiling water, and charcoal filter it, recovering a couple of more small crops of crystals if possible. Finally the crystals will begin to look funny, although they are clean. This is indicative that the less soluble mescaline has almost all crystallized out and the other alkaloids are starting to crystallize. These alkaloids are interesting and worth saving, but are non-hallucinogenic and are slightly poisonous. Many tailings may be saved and combined. Chromatography will separate them and recover more mescaline. Re-extraction from an alkaline solution may help get rid of $Na_2SO_4$ excesses. This is the complete purification process.

The final product is mescaline hemi-sulfate $2H_2O$. If pure mescaline, a clear corrosive oil, is desired, the procedure is simple from this point. Dissolve the pure hemi-sulfate in a 10% NaOH solution and extract with benzene. Evaporation in vacuo gives pure mescaline, but exposure to $CO_2$ in the air leads to the formation of mescaline hemicarbonate. Careful titration of the mescaline-benzene solution with any desired acid and careful, slow evaporation of the resulting aqueous extract yields pure crystals of the salts of that acid.

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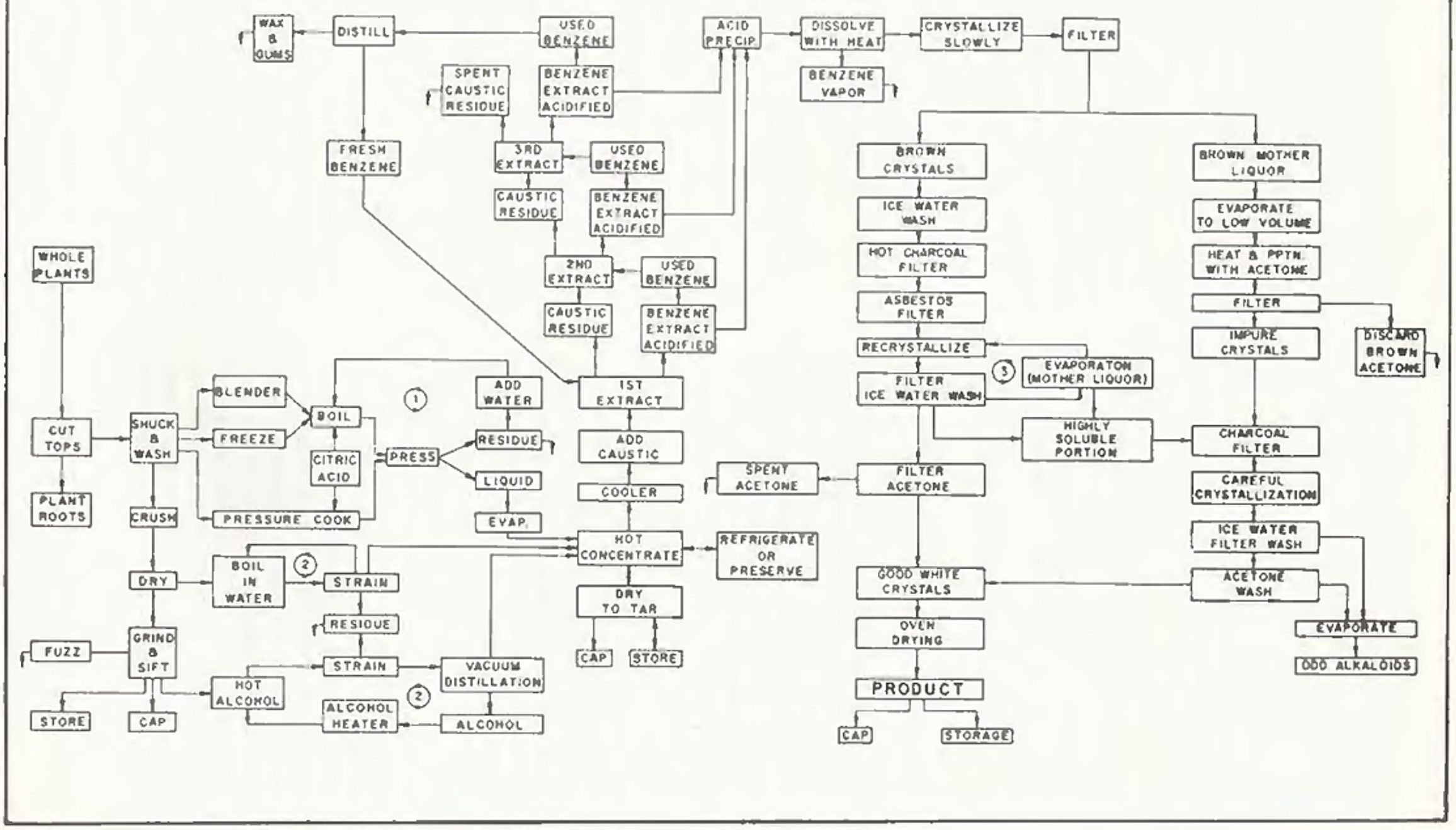
# peyote extraction chart

COMPLETE WITH ALTERNATE PROCEDURES



## MESCALINE SYNTHESIS #1

Mescaline, the alkaloid found in Lophophora williamsii cacti has been structurally defined and synthesized for nearly 50 years. The following procedure gives one of the first methods of synthesis slightly modified. Many other routes to phenylethylamine derivatives are possible, but this variation gives a much better yield. In most procedures the starting material is the same; 3, 4, 5, trihydroxy benzoic acid, more commonly known as gallic acid.

### STEP I – 3, 4, 5, Trimethoxy Benzoic Acid

[1]One g of $NaHSO_3$ is added to a 1000ml RB flask containing 500ml cold $H_2O$ with 80g (2 Moles or 110 ml 50% sol) NaOH and the flask is tightly stoppered and stirred with a magnetic stirrer for 10 minutes. To this solution is added rapidly 50 g (0.266 moles) of gallic acid. Gallic acid in NaOH solution oxidizes quickly with free oxygen to a brown compound, hence the $NaHSO_3$. The flask is re-stoppered quickly and stirred until all of the acid is dissolved. 89 g [67ml of dimethyl sulfate (0.71 moles] is then added and the flask is stirred for 20 minutes, being cooled by means of cold water in order that the temperature does not rise above 30 to 35°. (Dimethyl sulfate fumes are very toxic.) Occasionally the stopper is raised to release any pressure. A second portion of 89 ml dimethyl sulfate is then added and stirring continued for 10 minutes longer. During this addition the temperature may rise to 40-45°. Failure of the temperature to rise during the reaction may mean a delayed reaction which will begin almost explosively when reflux is begun.

The flask is then fitted with a reflux condenser and the contents boiled for 2 hours. In order to saponify the small amount of ester which is produced, a solution of 20% NaOH in 30ml $H_2O$ (12 ml 50% made up to 30ml) is added and boiling continued for 2 additional hours. The reaction mixture is then cooled and acidified with dilute HCl; the precipitated trimethoxy gallic acid is filtered with suction and washed well with cold water. The product which melts at 157-160° is sufficiently pure for most purposes. 50-52 g (89-92% yield).

Further purification is achieved by recrystallization from 2 liters of boiling water with the use of decolorizing charcoal; the filtration being carried out in a steam jacketed funnel. In this way 41-42 g of colorless needles melting at 167° is obtained.

### STEP II – 3, 4, 5, Trimethoxy Benzoic Chloride

[2]To 500 g of trimethoxy benzoic acid, add 285ml freshly distilled thionyl chloride and heat the mixture two hours in a water bath. The mixture is distilled under reduced pressure using glass connections at all joints. 510 g 3, 4, 5 trimethoxy benzoyl chloride boiling at 185° (18mm) are obtained. Yield 93% MP 83-84°.

### STEP III – 3, 4, 5, Trimethoxy Benzaldehyde

The Rosenmund reduction of acid chlorides to aldehydes is used with this compound, using no catalyst poison.

[3]To prepare the Pd-$BaSO_4$ catalyst, make a suspension of 1.7 g dry palladium chloride in 100 ml $H_2O$ containing 1ml con HCl, and heat upon a steam bath or allow to stand for several days until a clear dark red solution is obtained. A solution of 15 g anhydrous $Na_2SO_4$ in 200ml $H_2O$ is added in the course of 5 minutes to a mechanically stirred solution of 21 g $BaCl_2 \cdot 2H_2O$ in 200ml $H_2O$ at 70°. The precipitate is washed by decantation with hot water until the washings do not give a precipitate with $AgNO_3$. The $BaSO_4$ is then sus-

pended in 300ml $H_2O$ containing 1 ml of 40% formaldehyde. Ths suspension is heated to 80° and the solution of palladium chloride is added. The well-stirred mixture is neutralized to litmus by the addition of 1 N NaOH solution over a period of 15 to 30 minutes. Heating and stirring are continued for 20 minutes after a weakly alkaline reaction has been observed. The gray precipitate is filtered by gentle suction and is dried in a desiccator over calcium chloride. The dry catalyst contains about 5% palladium.

The hydrogen used must be free from oxygen. This is accomplished by passing electrolytic hydrogen over a heated copper coil. Drierite suffices to remove any $H_2O$. Sodium distilled xylene is put in the reaction vessel with the catylst and refluxing is begun with a hydrogen flush to remove latent water vapor. [2]To the slurry of 1000ml boiling xylene in which has been suspended 60 g of Pd-$BaSO_4$ catalyst, add 200 g 3, 4, 5 trimethoxy benzoyl chloride. This mixture is heated on an oil bath maintained at 150° and a vigorous stream of $H_2$ is introduced into the boiling solution. Fit the reflux condenser with a drying tube and from there, bubble the gas through a water solution. Continually titrate the HCl being given off until no more is evolved. This takes from 60-80 hours. The solution is filtered and precipitated with a solution of $NaHSO_3$.[3] The aldehyde bisulfite precipitate is filtered and washed with ether. The compound is decomposed by boiling with excess $Na_2CO_3$ and the aldehyde is extracted with ether. Yield 120 g (70.6) MP 74°.

**STEP IV – 3, 4, 5- Trimethoxy**
**B.Nitrostyrene**

A solution of 40ml nitromethane and 100 g 3, 4, 5 trimethoxy benzaldehyde in 200ml alcohol is cooled to 0° and while it is stirred mechanically, there is introduced a solution of 45 g pure KOH in 45ml $H_2O$ and 90ml methanol at the rate of about one drop per second, care being taken that the temperature does not rise. 15 minutes after the addition is completed, the solution is poured into 500ml con HCl mixed with sufficient ice to assure its presence throughout the slow addition and to maintain a temperature of -10°. The precipitated 3, 4, 5, trimethoxy B nitrostyrene is separated by filtration and washing and may be purified by crystallization from 700ml alcohol. The pale yellow plates which melt at 120-121° are obtained in a yield of approximately 78%.

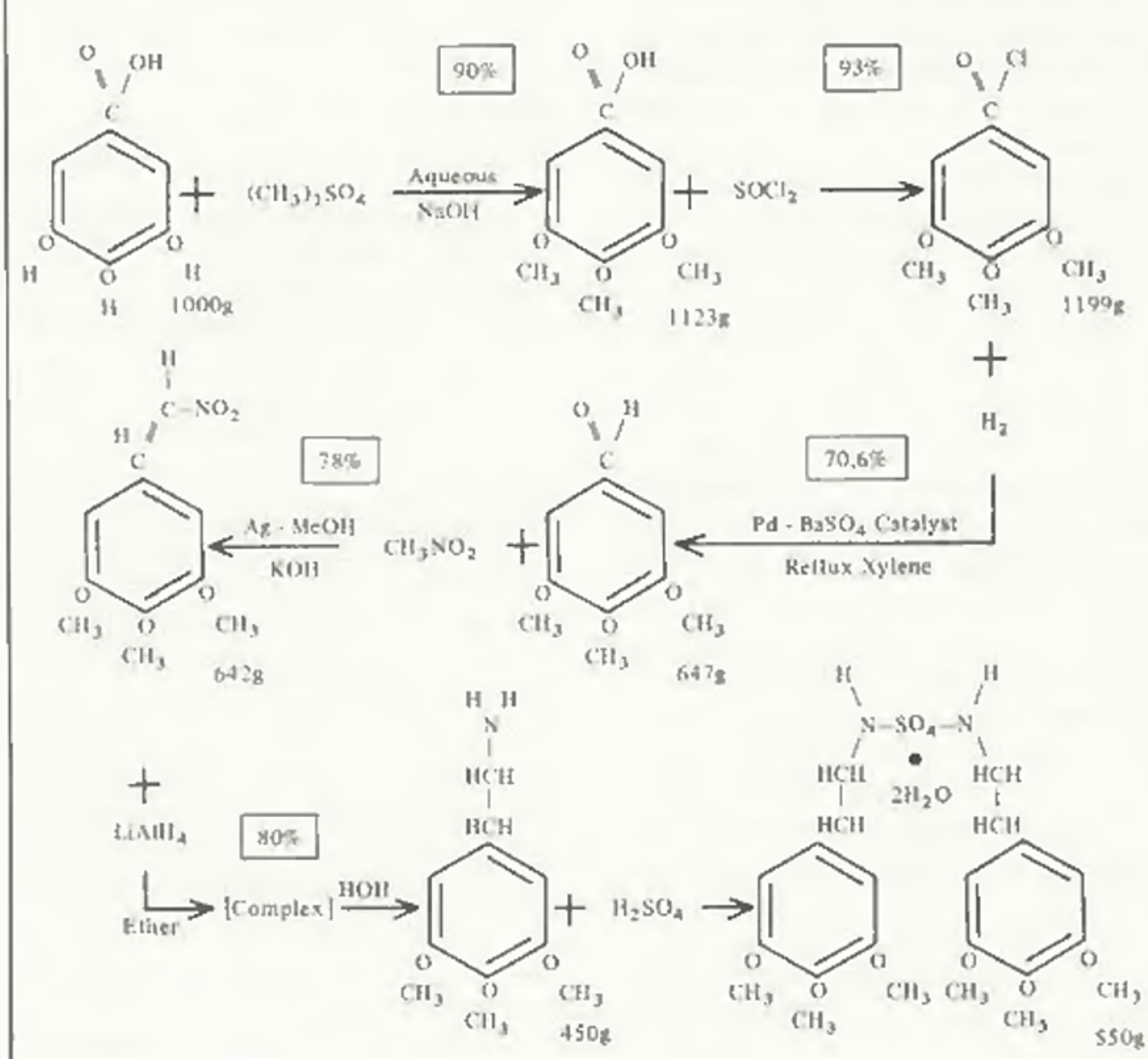
**STEP V – 3, 4, 5- Trimethoxy**
**B Phenylethylamine**

[4]To 1000ml anhydrous ether in a 2000ml RB flask is added 57 g $LiAlH_4$ while stirring vigorously with a magnetic stirrer. (It is a good idea to use a safety shield while working with $LiAlH_4$.) Place 72 g 3, 4, 5 trimethoxy B nitrostyrene in the thimble of a Soxhlet extractor and attach the extractor to the flask with a condenser above. Heat the flask until ether boils gently and the extractor cycles. Attach a cold trap and a drying tube from the top of the condenser to stop ether loss and hydration. [5]Reflux and react for about 59 hours, watching the contents of the extractor. The operation may be interrupted at any time as long as water vapor does not get in. If another solvent, such as tetrahydrofuran or dioxane can be found which combines solubility and inertness to $LiAlH_4$ with solubility to the nitrostyrene, the Soxhlet extractor would be unnecessary, time would be reduced to about 2 hours, and the nitrostyrene could be added in solvent solution.

After the reduction is complete cool the flask in an ice-bath, stirring vigorously, and cautiously hydrolyze the contents by adding chips of ice through the condenser (extractor removed). When the reaction ceases, allow to sit for 2 hours. Add 500 ml 10% NaOH and remove the ether solution. Extract 3 times with 2N $H_2SO_4$ using a magnetic stirrer. At pH 7 (on pH paper), pour off the solvent and recrystallize the mescaline hemi-sulfate di-hydrate from boiling water. Yield, 74-86% MP 183-186°.

## REACTIONS

Starting with 1000 g gallic acid, each yield given as reported in the literature:





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agent, the ether solution is warmed on a steam bath and the ether is removed with a stream of air. On chilling, the residue yields scale-like crystals. Recrystallization from ether gives rectangular prims; yield, 2.5 g (27%); MP 76-77°

### STEP VI – Mescaline

In 150 ml of anhydrous ether is suspended 0.85 g of lithium aluminium hydride powder. With stirring, 2.0 g of 3, 4, 5, trimethoxy phenylacetonitrile in 150 ml of anhydrous ether was added during the course of 15 minutes. After 15 minutes stirring, 10 ml of ice-water is dropped in carefully. Then a mixture of 10 g of sulfuric acid in 40 ml of ice-water is added at a moderate rate. The aqueous layer is separated and treated with con sodium hydroxide. The brown oil is extracted with three portions of 30 ml each of benzene. The combined benzene extracts are titrated to neutrality with 10% sulfuric acid. The aqueous layer is filtered through charcoal, heated, and cooled slowly to yield colorless needles which soften at 172° and melt at 182°.

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## MESCALINE SYNTHESIS – # 1 & # 2 COMBINED
### An Intermediate Step

A good method using the best halves of both procedures involves the first four steps of the second method, using the Sommelet reaction to obtain the aldehyde, and finishing with the first method; steps four and five.

### Intermediate Step – 3, 4, 5- Trimethoxy Benzaldehyde

To a solution of 100 g 3, 4, 5, trimethoxy benzyl chloride in 500 ml, 60% aqueous ethanol, is added 80 g of hexamethylenetetramine. This reagent is easily prepared by adding an excess of con ammonia to a formalin solution and evaporating the solution to obtain the solid hexamethylenetetramine. Reflux this mixture for one hour and purify the aldehyde as in Step III, Synthesis #1. Yield 70-80%; 70 g. Recover any un-reacted 3, 4, 5, trimethoxy alcohol or over-oxidized acid using techniques as in purification of those intermediates.

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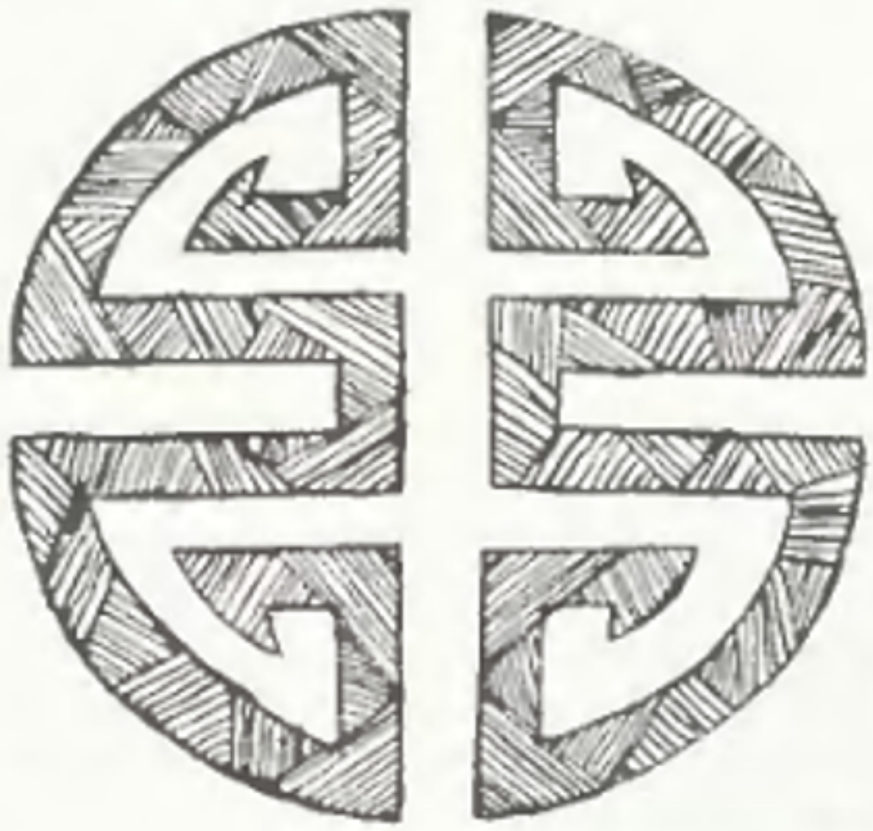


## MESCALINE SYNTHESIS #2

This method of synthesis is included because it is commonly used and is a little easier than the first one. The advantage in the first synthesis is an overall yield of nearly 45%, while this procedure nets only about 5% theoretical yield.

### STEP I – 3, 4, 5- Trimethoxy Benzoic Acid

Use the procedure for Step I, Synthesis #1.

### STEP II – Methyl Ester of 3, 4, 5- Trimethoxy Benzoic Acid

To a solution prepared from 100 g of 3, 4, 5, trimethoxy benzoic acid (0.47 Mole), 20 g sodium hydroxide, 55 g of sodium carbonate and 300ml of water is added, with stirring, 94 ml of dimethyl sulfate (0.94 Mole) during the course of 20 minutes. The reaction mixture is refluxed for one half hour. The crude ester (65 g or 61% yield) precipitates from the cold mixture. From the filtrate, 38 g of starting material is recovered upon acidification with dilute hydrochloric acid. The ester is further purified by solution in the minimum amount of methanol and treatment with norite. Usually it is necessary to repeat this treatment to obtaina colorless crystalline product that melts at 80-82°C..

### STEP III – 3, 4, 5- Trimethoxy Benzyl Alcohol

To a suspension of 4.6 g (0.12 Mole) of lithium aluminum hydride in 200ml anhydrous ether is added, in the course of 30 minutes, a solution of 22.6 g (0.1 Mole) of the methyl ester of 3, 4, 5, trimethoxy benzoic acid in 300ml of dry ether. The solid which forms is carefully decomposed, first with 50 ml of ice water, cautiously added in small portions. After decantation of the ether, 250 ml of ice-cold 10% sulfuric acid is added. The product is extracted with 150 ml of ether. The combined ether extracts after drying over sodium sulfate, are freed of ether and the residue distilled; b.p. 135-137° (0.25mm); yield 14.7 g (73%). Again, some 3, 4, 5, trimethoxy benzoic acid may be recovered from the extraction residue.

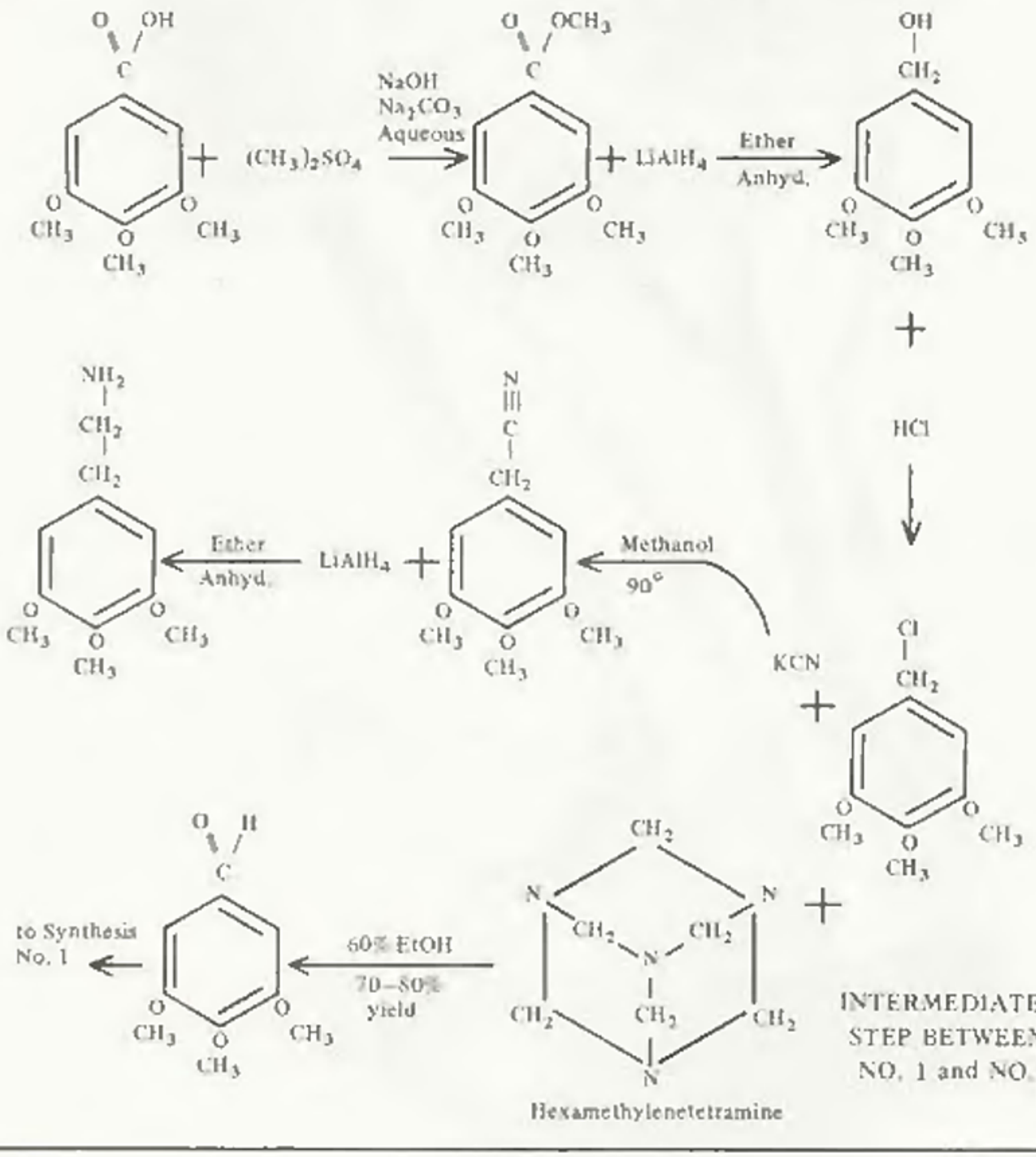
### STEP IV – 3, 4, 5- Trimethoxy Benzyl Chloride

A mixture of 25 g of 3, 4, 5, trimethoxy benzyl alcohol and 125 ml of ice-cold con hydrochloric acid is shaken vigorously until a homogenous solution is obtained. In a few minutes a turbidity develops, followed by a heavy precipitation of gummy product. After four hours and dilution with 100 ml of ice-water, the aqueous layer is decanted and extracted with three 50 ml portions of benzene. Then the gummy organic residue is dissolved in the combined benzene extracts. The benzene solution is washed with water and dried over sodium sulfate. The benzene solution is then transferred to a distillation flask and the benzene is removed under diminished pressure. The red semi-solid residue is suspended in a small amount of ice-cold ether and filtered through a chilled funnel. The crystalline product may be further purified, and after four recrystallizations from benzene, colorless needles are obtained; MP 60-62°. This product should not be stored very long as it may hydrolyze to a slight extent.

### STEP V – 3, 4, 5- Trimethoxy Phenylacetonitrile

A mixture of 9 g of potassium cyanide in 35 ml of water and 60 ml methanol and 9.7 g of 3, 4, 5, trimethoxy benzyl chloride is heated for ten minutes at 90°. The solvents are partially removed under diminished pressure. The residue is then extracted with 90 ml of ether in three portions. The combined extracts are washed with water and dried over sodium sulfate. After removal of the drying

# mescaline synthesis-2



PSILOCYBIN CULTURE

The purpose of these instructions is to provide the details of producing psilocybin from a natural source. The traditional mushrooms are difficult to grow, but the fungal mycelia or thread-like portions of the same organism may be grown in culture, producing considerable psilocybin.

It is important in working with fungi to use pure culture technique to prevent the vigorous wild molds taking over the slower growing psilocybe fungus. Pure culture technique is treated in detail in any good microbiological manual. Better yet, find someone who has had bacteriological training to help you learn how to transfer cultures with an innoculating loop.

The psilocybe fungus must be pure-cultured prior to any other operations. Make a sterile water solution of the spore dust and plate it out in dilutions on potato dextrose yeast agar in Petri dishes. After growing several days some of the plates should show signs of the white mycelial mats of the psilocybe fungus. Obvious wild molds must be discarded and the selected culture growths may be tested by reculturing in PDY broth and testing a methanol extract of the fungus with the Keller-Reagent (see note below).

Make agar slants for storing cultures by filling 6 inch by ½ inch screw cap tubes one-third full of melted PDY agar. Autoclave and let cool at an angle so the gelled agar makes a slanted surface in the tube. Innoculate the slants with the cultures from the Petri dishes, taking care to keep everything free from external contamination. The lightly capped tubes are stored at room temperature until a mycelial mat has grown. Screw the caps tight and store in a refrigerator. These will keep about a year before they will need reculturing.

The main culture medium is a liquid, prepared according to formulae listed below. Culture jars may consist of various-sized mason jars with covers made from heavy-gauge aluminum foil. In as much as media are prepared to culture *Psilocybe* mycelia to the extinction of all other organisms, it is necessary to sterilize the culture jars with the medium in them. Sterilization is best accomplished by placing the covered jars (no more than half full of medium) into a canning pressure cooker containing a little water. A temperature of 250°F is most easily maintained if the pressure cooker has a gauge giving temperatures at different pressures. After 15 to 20 minutes, cut off the heat, but keep the cooker sealed, as sudden loss of pressure will cause the medium to boil over. If the temperature goes too high or cooking is too long, some of the sugars will begin to carmelize. This reaction renders the medium unfit for growing the fungus since caramel slows growth.

## MEDIA FORMULAE

### Potato Dextrose Yeast Agar

Wash 250 grams potatoes (do not peel).
Slice 1/8 inch thick.
Wash with tap water until the water is clear
Drain, rinse with distilled water
Cover with distilled water and cook until tender
Drain liquid through flannel cloth or several thicknesses of cheesecloth into a flask or jar
Rinse potatoes once or twice with a little distilled water
Keep liquid and throw potatoes away — add enough distilled water to make up one liter of liquid

Bring liquid to a boil and add:

15 grams of agar — stir until dissolved (watch carefully or it will boil over - best to use an open stainless steel pan)

10 grams of dextrose

1.5 grams of yeast extract

While liquid is hot, distribute into desired containers
Autoclave for 15 minutes at 250°F (about 15 lbs. pressure)

PDY broth is made in the same way omitting the agar.

rye grain medium:

for ½ pint jars:

50 grams rye grain (whole)
80 ml. water
1 gram chalk (Calcium carbonate)

for pint jars:

100 grams rye grain
160 ml. water
2 grams chalk

for quart jars:

225 grams rye grain
275 ml. water
4 grams chalk

Note: the grain medium may seem to be a bit dry at times; if so, a few ml. of sterile water may be added.
The rye medium is also used as a "spawn" for innoculation of horse manure compost as is done in commercial mushroom culture. Methods for growth of *Psylocybe* on compost have been worked out, but require access to a commercial source of compost, or know-how of preparing small amounts of compost. *Ps. mexicana* does not fruit on compost, but *Ps. cubensis* will.

After preparation and sterilization, keep the media at room temperature for three days without opening, as a check to see if the sterilization technique was effective. Any growth or scumminess indicates unwanted growth and that medium must be discarded.

These large containers of broth may now be innoculated carefully with loops of whitish mycelia taken from the pure stock cultures, using pure culture technique. Keep covered and incubate at 70-75°F for 10-12 days. Temperature is important as the fungus produces psilocybin poorly at higher temperatures and grows poorly at lower temperatures. Ideally, harvesting is done four days after all the sugar in the medium has been used by the fungus. This may be followed with the use of a simple saccharimeter, if desired. Otherwise, trial and error will establish the optimum incubation time which will produce the best yields.

The culture contains a mat of fibrous mycelia at the end of the growth period. No mushrooms or carpophores are produced, but these are not essential to psilocybin production since the mycelia contain an equal percentage. Filter the medium through a flannel cloth, collect the matted mycelia from the cloth and dry it in a dryingoven at less than 200°F. A kitchen oven may be used if the door is kept partly ajar and the temperature is watched closely. The mycelium residue is powdered and extracted three times with methanol and the methanol carefully evaporated. A hair dryer does this nicely, but the fumes from methanol are terribly poisonous and the area must be well ventilated. Take care to remove all of the poisonous solvent from the psilocybin residue.

Practice, with the help of a competent bacteriologist, will improve yields. If possible, have an expert in mycology isolate the pure psilocybe fungi from the dried mushrooms.

*Psilocybe cubensis* grows and fruits readily on the potato dextrose yeast (PDY) agar or on sterilized grain such as rye; *Psilcoybe mexicana* fruits only on PDY agar.

See the U.S. Pharmacopoeia for the Keller-Reagent, a solution of ferric chloride in glacial acetic acid used as a general test for alkaloids. (Not specific for psilocybin.)

For illustrations of the fruiting bodies (carphophores) of Psilocybe mexicana see Compt. Rend. Acad. Sci. 246: 1346-1351 (1958).

For the extraction method see Experimentia 14: 107 (1958).

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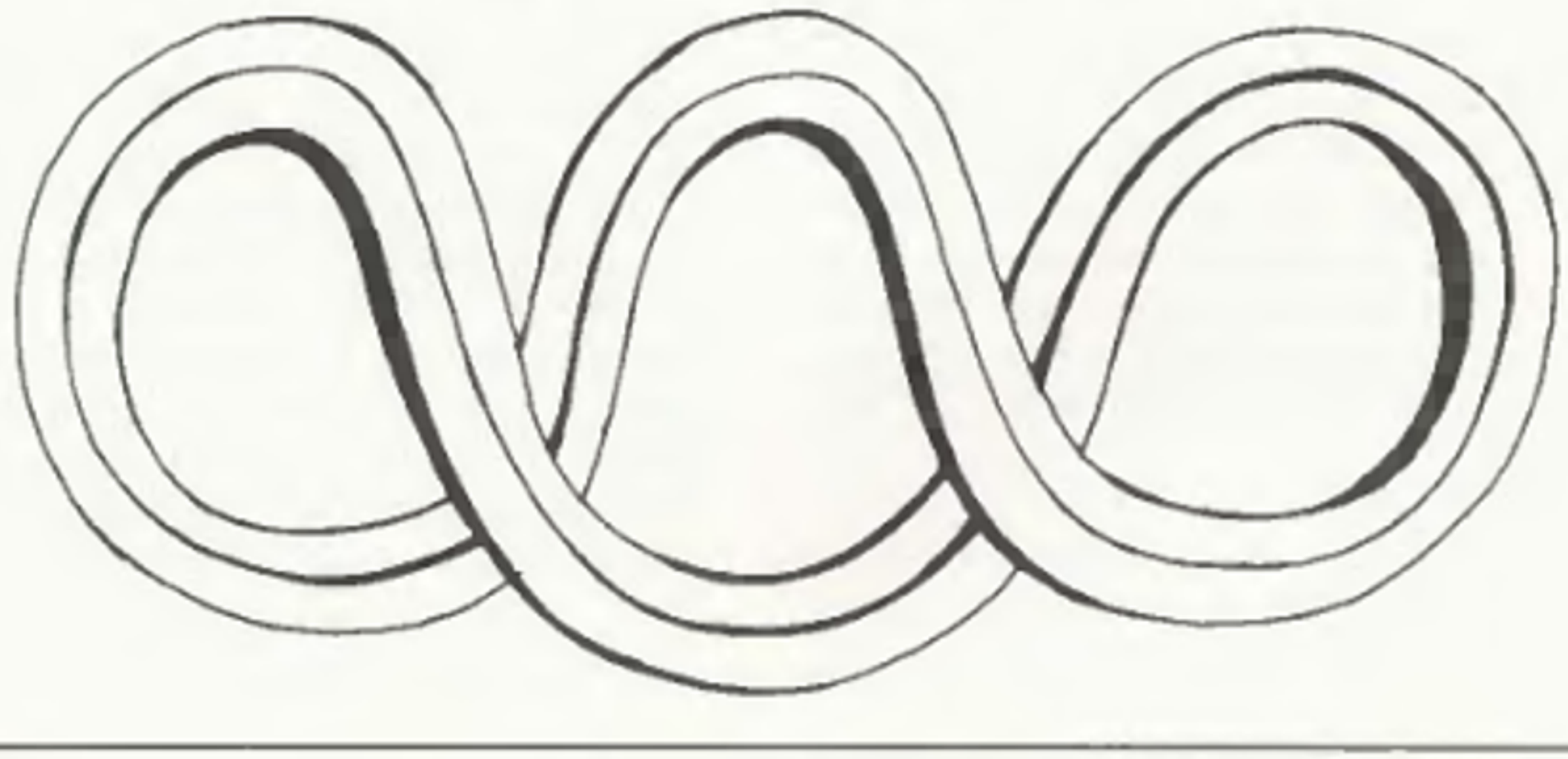

## SYNTHESIS OF PSILOCYN

### Introduction

This synthesis is designed to produce psilocyn from obtainable materials, but is very long and involves many steps as a consequence.

### STEP I

6-nitro ortho-toluidine is prepared by reduction of an ammoniacal-alcohol solution of 2, 6-dinitro toluene using hydrogen sulfide or ammonium sulfide.

Hydrogen sulfide is a deadly, foul-smelling gas which must be handled out of doors on a windy day. This reagent may be purchased as a compressed cylinder gas or may be generated by action of acid on ferrous sulfide or water on aluminum sulfide. Ammonium sulfide solution is much safer and easier to use and is produced in the reaction anyway, even if the gas is used.

20 g of 2, 6-dinitro toluene is mixed with 60 ml of 93% ethanol and 50 ml of concentrated ammonium hydroxide. The mixture is warmed and stirred while hydrogen sulfide is bubbled through the solution for one hour. If ammonium sulfide is added, the ammonium hydroxide is not necessary, but the sulfide solution must be added dropwise over the period of an hour. The solution is transferred to a casserole and the alcohol evaporated. The residue is acidified with HCl and is extracted with that hot acidic solution, the toluidine being only partly soluble in cool water. Filter the solution cool and make basic with ammonium hydroxide. Filter and wash with water; recrystallization is not necessary. The base melts at 91.5°; yield, 96%.

### STEP II

25 g of 6-nitro ortho toluidine is dissolved in a mixture of 50 ml con sulfuric acid in 800 ml of water. 12.5 g of sodium nitrite is dissolved in water and dropped in while the whole thing is cooled in an ice-bath for two hours. The whole reaction may conveniently be carried out in a gallon jug if care is taken to protect the glass from thermal shock. React the solution overnight in a refrigerator to take care of insoluble reactants. To the jug containing the filtered cooled solution add 2167 ml water with 250 ml con sulfuric acid and warm in a large kettle full of warm water gradually brought to a boil while the jug is in it. After nitrogen bubbles cease to be evolved, fill the jug nearly to the top and skim any residue from the surface. Cool the mixture slowly to near 0° and filter the crystalline nitrocresol from the solution. Ether extraction of the solution will yield another gram of product. Yield, 22.2 g.

### STEP III

Dissolve 77 g 6-nitro ortho cresol in a solution of 20 g sodium hydroxide in 200 ml water in a roundbottom flask. A trace of sodium sulfite will remove any dissolved oxygen which might oxidize the sodium cresol. Controlling the temperature with an ice-bath, add cautiously 63 g (47.8 ml) of cold dimethyl sulfate, making sure that the cresol is completely dissolved first. Extend the addition to the period of one hour, vigorously swirling the contents after each addition. After the addition is complete, continue swirling while the flask warms up to room temperature and warm the flask further to 100° in a water bath for an hour. The product separates out as a dark oil in the bottom which may be washed with hot water. Cool and extract the dark oil with benzene or ether. Dry the solution over anhydrous sodium sulfate.

STEP IV

2-nitro 6-methoxy toluene is converted to 2-nitro 6-methoxy phenyl pyruvic acid by reaction in potassium ethylate and diethy oxalate.

Potassium metal (7.8 g) is shaken under xylene at 100° until it is reduced to a fine suspension which readily settles as the liquid cools. After the xylene has been decanted, the metal is washed twice with anhydrous ether (170 g or 238 ml), leaving the last portion on the powdered metal. This operation is very dangerous due to the reactive nature of potassium metal and even contact with damp air is likely to start a fire. Absolute ethanol is cautiously added to the ether-potassium slurry care being taken not to add so much at once that the ether boils too vigorously. After standing one-half hour, the liquid is filled with a crystalline precipitate of potassium ethylate. Diethyloxalate (29.2g or 27 ml) is now added. The solid ethoxide dissolves to a clear orange solution with the production of sufficient heat to cause the ether to boil. To this clear solution is added 12.5 g of 2-nitro 6-methoxy toluene, and the reaction mixture immediately turns red. After this has been gently heated under reflux at 35-38° for 18 hours. the potassium derivative separates as a dark red, rather gummy precipitate, which is extracted with water and the red alkaline solution filtered, washed with a little ether to remove unreacted nitro methoxy toluene, and acidified with dil HCl. Upon blowing air through the liquid to remove dissolved ether, 2-nitro 6-methoxy phenyl pyruvic acid, separates out as a brown oil which may solidify slowly. This is collected and dried in air.

STEP V

Crude 2-nitro 6-methoxy phenyl pyruvic acid (23.9) g is dissolved in ammonia (140ml of .88° ammonium hydroxide made up to 200 ml with water) and a hot water solution of ferrous sulfate (180 g of hydrated crystals in 200 ml water) is added to the reddish-brown, alkaline solution. Reduction is instantaneous and the black mixture is heated on a water-bath for half an hour with frequent shaking, and then gently boiled for the same length of time. After cooling, the black sludge of ferric hydroxide is filtered off using some filter aid such as Celite or asbestos pulp to keep the filter from becoming stopped up with the slimy black precipitate. Wash the precipitate with dilute hot ammonia until a portion of the filtrate is no longer cloudy when acidified with HCl. Concentrate the filtrate nearly to dryness, acidify with HCl and extract with acetone. Evaporate the acetone, dissolve the residue in a small portion of con ammonia and re-acidify with HCl. The 6-methoxy indole 2-carboxylic acid separates out as a dirty white, sandy precipitate, which is collected and dried at 100°. Melting point 235°.

STEP VI

3 grams 6-methoxy indole 2-carboxylic acid is decomposing by heating at 245-250°F for one hour and the indole distilled at 181-183° at 24 mm Hg. A yellow crystalline mass collects in the receiver and is powdered and warmed with dilute potassium carbonate to remove unchanged acid. Filter and wash with dilute potassium carbonate and then water, saving the filtrate to be acidified for recovery of residual acid. The precipitate of 6-methoxy indole is dried in a vacuum desiccator over con sulfuric acid, and may be recrystallized from petroleum ether if desired.

STEP VII

Proceed, using the processes for DMT, using 75 g of 6-methoxy indole in place of 70 g indole. For the lithium aluminum hydride reduction, use 22.7 g of the IOC methoxy derivative. The final product will be 6-methoxy DMT which may be used in that form or

converted to 6-hydroxy DMT (psilocyn) by warming with a solution of hydriodic acid, followed by extraction with ether, from the solution made basic with ammonium hydroxide. Psilocybin is difficult to produce and the body must convert it to psilocyn before it is absorbed, so it has no advantage.


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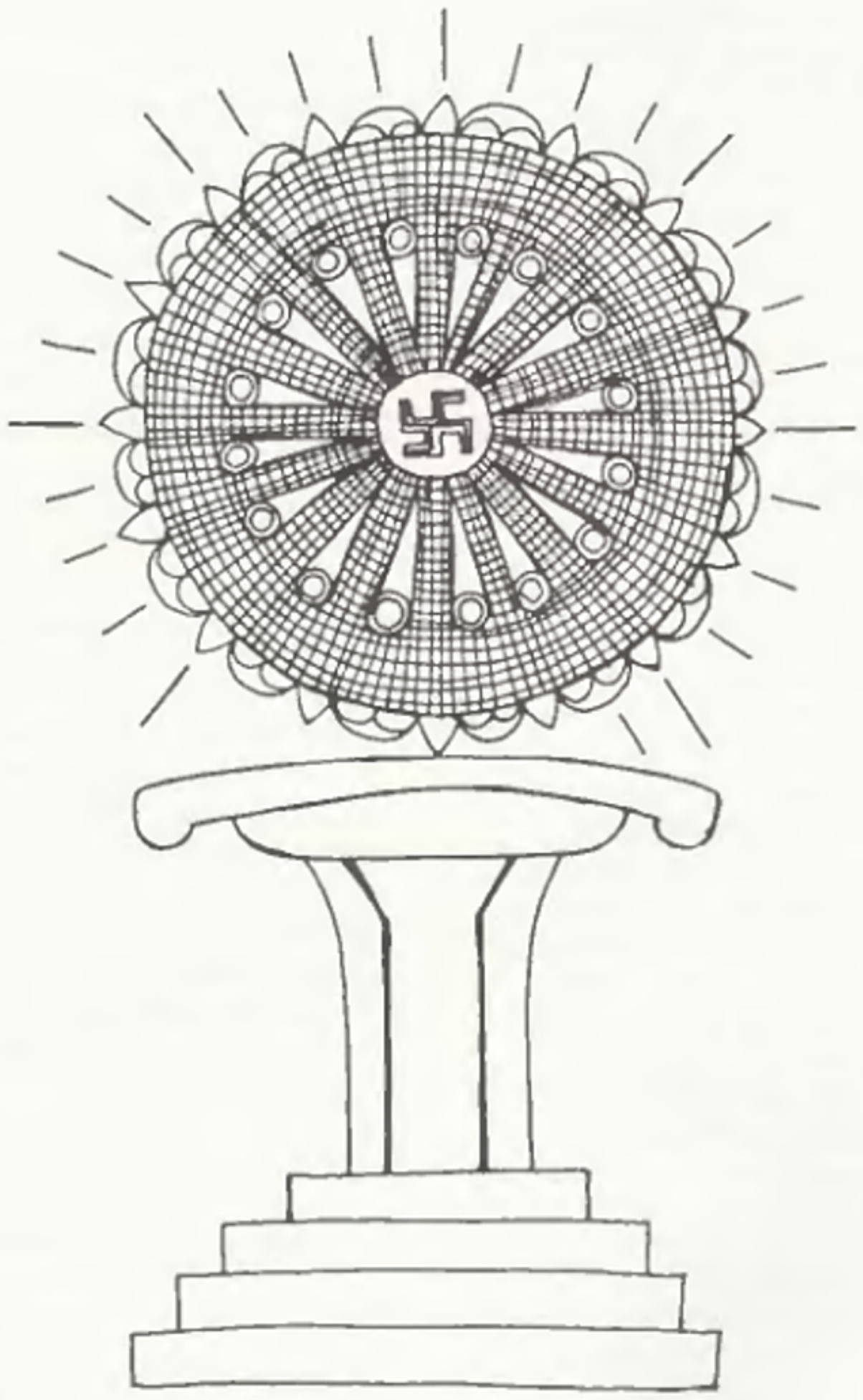
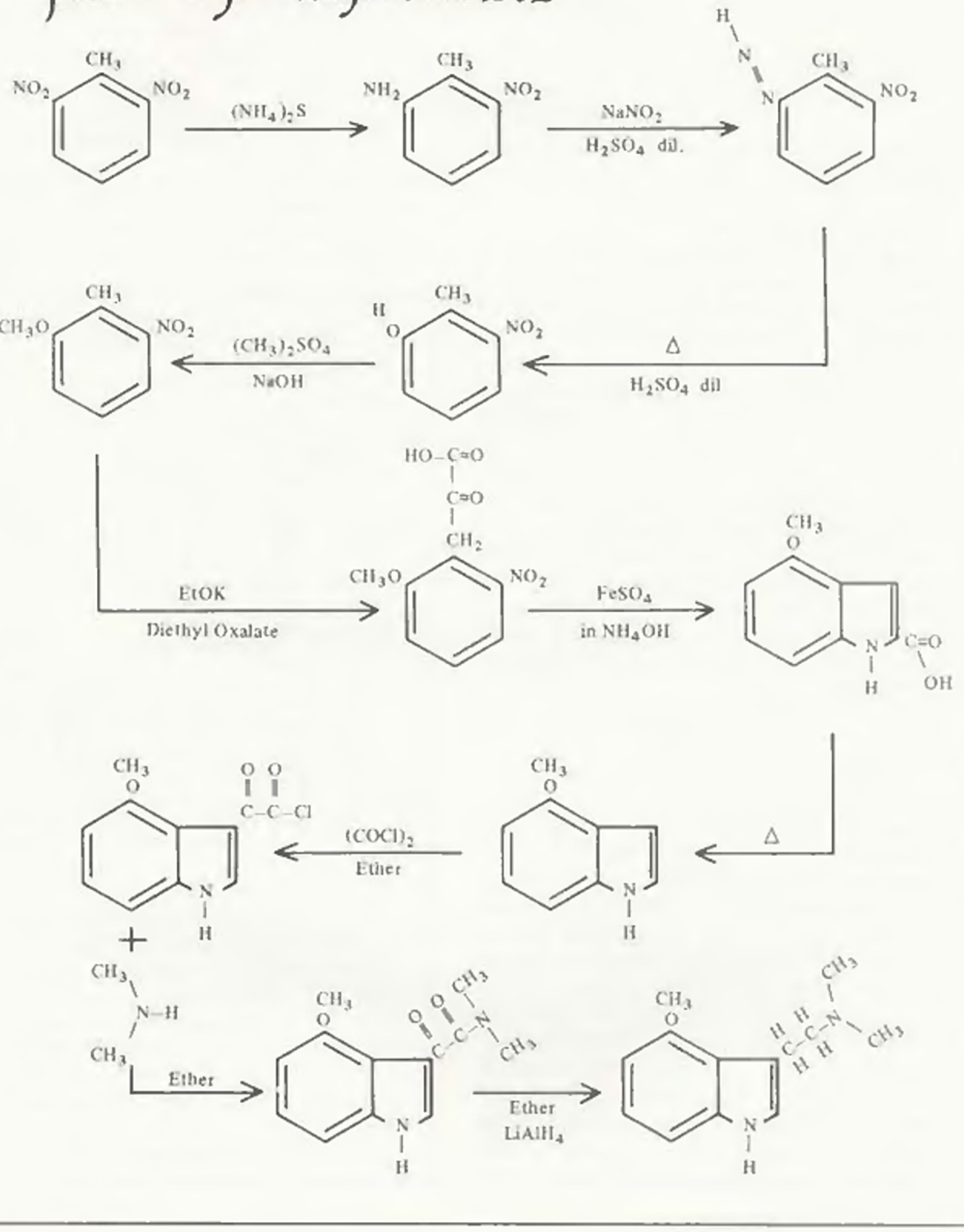


# psilocyn synthesis



## DMT Synthesis

### STEP I

Using an area of good ventilation or a fume hood, place a 1000 ml roundbottom flask in an ice bath using the set-up in Figure 1. Add 400 ml cold anhydrous ether to this flask, in which 60 g indole is then dissolved using the stirrer. To 100 ml anhydrous ether in a separatory funnel add 50 g of oxalyl chloride. Slowly drip this solution into the vigorously stirred indole solution over a period of 10 to 15 minutes. Continue stirring 10 minutes longer. Allow the precipitate to settle a few minutes and decant the liquid. Add anhydrous ether and mix well. Allow this to settle and decant the liquid again. When satisfied as to the purity of the precipitate, leave the golden precipitate in the flask for the next step which must follow immediately. Yield is approximately 100 g.

### STEP II

Dimethylamine reacts readily with indole oxalyl chloride. Use about 400 ml ice-cold anhydrous ether in the same 2 neck 1000 ml RB flask used in Step I, with the precipitate in it from Step I. Cool the ice bath further by using salt and ice. Estimate the weight of the precipitate and use 100 g indole oxalyl chloride. For this weight of IOC use two entire 50 g containers of dimethylamine since it will not keep if the container seal is broken. Cool the amine in container much below 0°C and dissolve 1 part amine in 3 parts anhydrous cold ether. Amine may be stored in this solution. For use, warm stock solution to room temperature and use the appropriate aliquot. Set up the entire apparatus the same as when adding the oxalyl chloride. Add the amine solution slowly to the IOC with vigor-

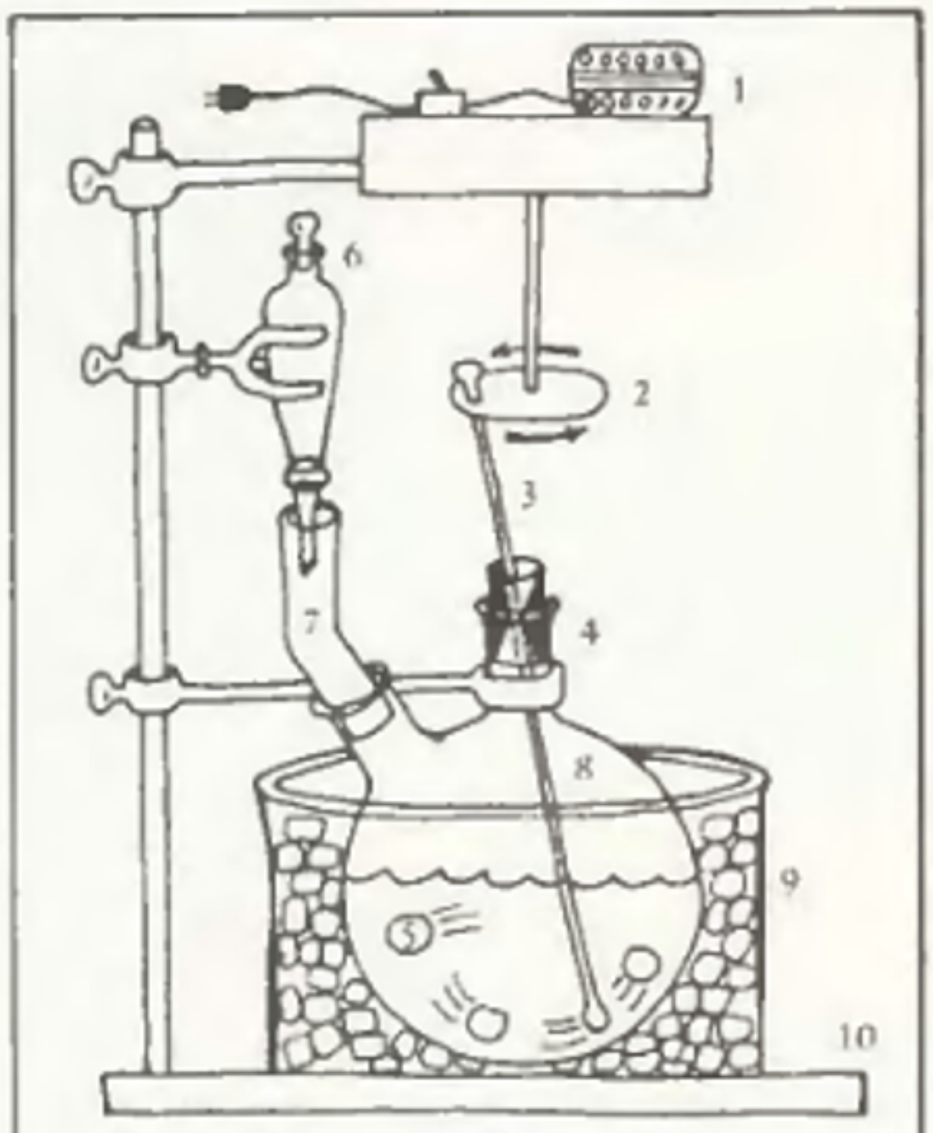



Figure 1

1. 100-500 RPM sparkless motor
2. Eccentric stirrer driver
3. Paddle Bladed stirring rod
4. Polyethelene stopper.
5. Weighted coated stir-balls
6. Dropping funnel (separatory)
7. 150° bent tube
8. 100ml 2 neck RB flask
9. Ice bath
10. Ring stand and clamps

ous stirring. Stir for ½ hour after the addition is complete, allowing the temperature to rise to ambient. Vacuum filter the precipitate using ether as a wash, followed by an ether-water wash. It is better to slurry the ether-water with the precipitate before filtering. Recrystallize from hot ethanol or from a 50-50 methanol-benzene mixture.

**STEP III**

Prepare apparatus as in Figure II. Prepare the indole glyoxal amide by melting and casting into sticks if ether is to be used as a solvent. Aluminum foil makes a good mold, for casting pieces which will fit through the condenser. Also a Soxhlet extractor may be used to add the crystals by slow solution into the ether. Tetrahydrofuran, if available dissolves IGA and the compound is added slowly in the solution form.

To a stirred mixture of 15 g $LiAlH_4$ in 300 ml anhydrous ether (or THF) slowly add the sticks (or solution) of IGA until 20 g in all have been added. Keep the rate of reaction at a reasonable level or boil-over may occur. Stir and reflux for 90 minutes after the addition is complete. Cool in an ice-bath and begin to cautiously hydrolyze the complex with chips of ice or a cold solution of methanol, added through the condenser.When there is no further reaction, add a few ml extra water and allow to settle finally and decant the clear liquid into an evaporating vessel. Filter the residue and wash several times with ether-methanol or THF-methanol. Evaporate the combined extracts and if necessary, seed the heavy syrup with crystals of DMT. With no seed crystals the product may take days or even weeks to crystallize. This crude product is adequate for smoking. In order to purify DMT, begin after the $LiAlH_4$ has been hydrolyzed with methanol. Add 500 ml satd. $Na_2SO_4$ solution, mix and filter. Wash with ether or THF and neutralize the filtrate with 0.1 N HCl. Extract with ether in a separatory funnel and neutralize the lower layer with 0.1 N NaOH, extracting this solution in turn with chloroform. The chloroform layer is dried over anhydrous $Na_2SO_4$, concentrated, and from it DMT crystallizes on addition of petroleum ether. The mother liquor can be chromatographed on an alumina column using benzene-methanol in a 99.8 to 0.2 ratio.

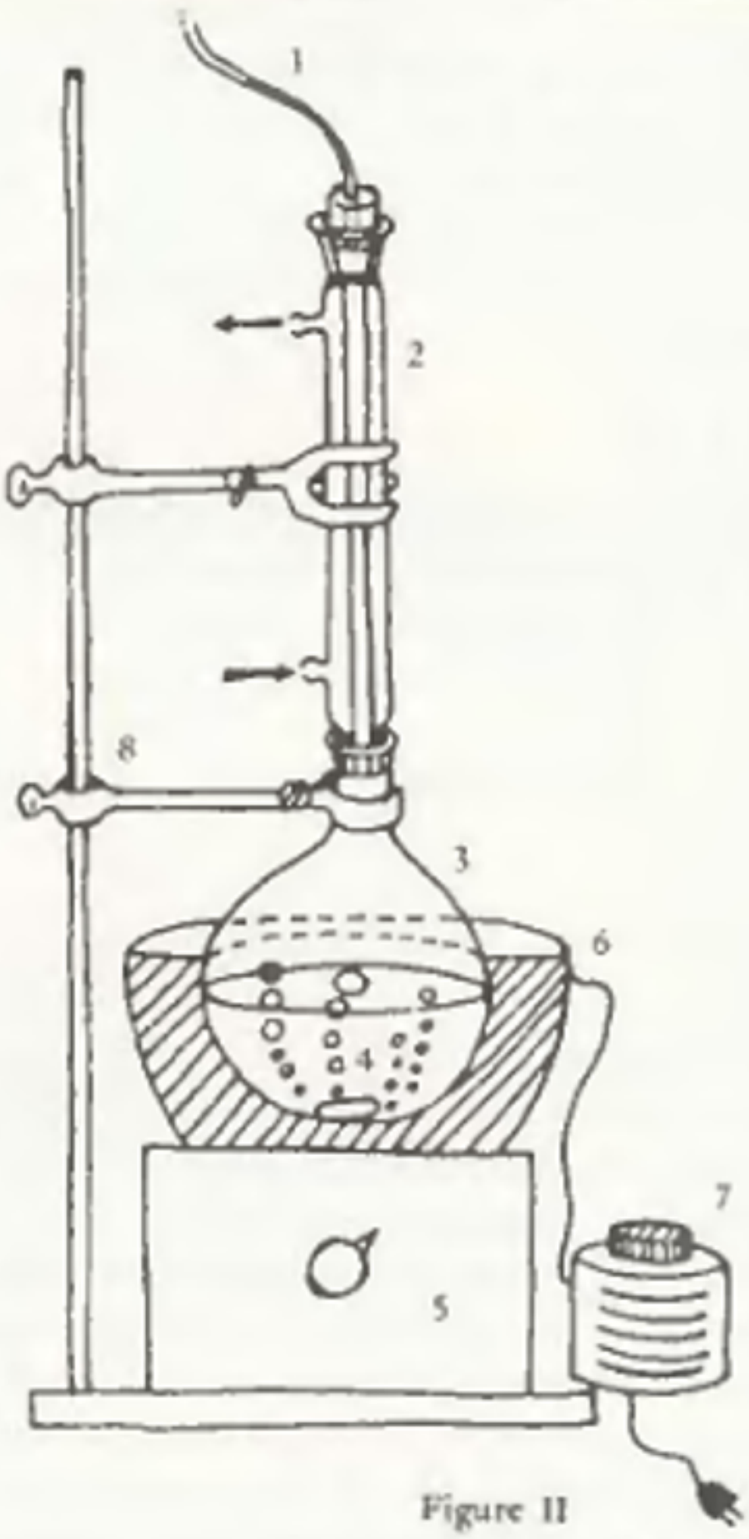



Figure II

1. Ether vent to outside
2. Condenser
3. 1000ml RB flask
4. Magnetic stir-bar
5. Magnetic stirrer
6. Heating mantle
7. Variac ( 6 & 7 replaced by ice-bath in last part)
8. Ring stand and clamps

DET

STEP I

Same as for DMT.

STEP II

Use 200 g diethylamine per 100 g IOC. Diethylamine is less volatile than dimethylamine, so cooling is not necessary, but the fumes are poisonous. Use the same procedure otherwise. Diethyl derivative is easier to work with.

STEP III

Use same procedure and equipment. Use 22 g indoleglyoxal diethylamide. The final product is also easier to purify.

NOTES

STEP I

Absolutely anhydrous ether is essential. A container that has been opened previously is no longer anhydrous. Where cooled reactions are necessary, remember that moisture is drawn to cold objects, and cold reagents, when left open or poured, become quite wet. This applies to the initial reaction in all three steps. A magnetic stirrer will not work for steps I and II. The vigorous wobble-stirrer has been found adequate to deliver the violent stirring needed, especially when several stirring balls are used in conjunction with the paddle-bar. Sparkless motors must be used around ether.

Oxalyl chloride is very toxic and ventilation or a fume hood must be used.

Water vapor hydrolyzes product I, producing a gummy dark-red mass. Proceed to step II as soon as possible.

STEP II

Refer to notes on anhydrous ether, stirrer, sparkless equipment, and ventilation in the notes for step I.

The color of the precipitate lightens somewhat as the amine is added to the compound I.

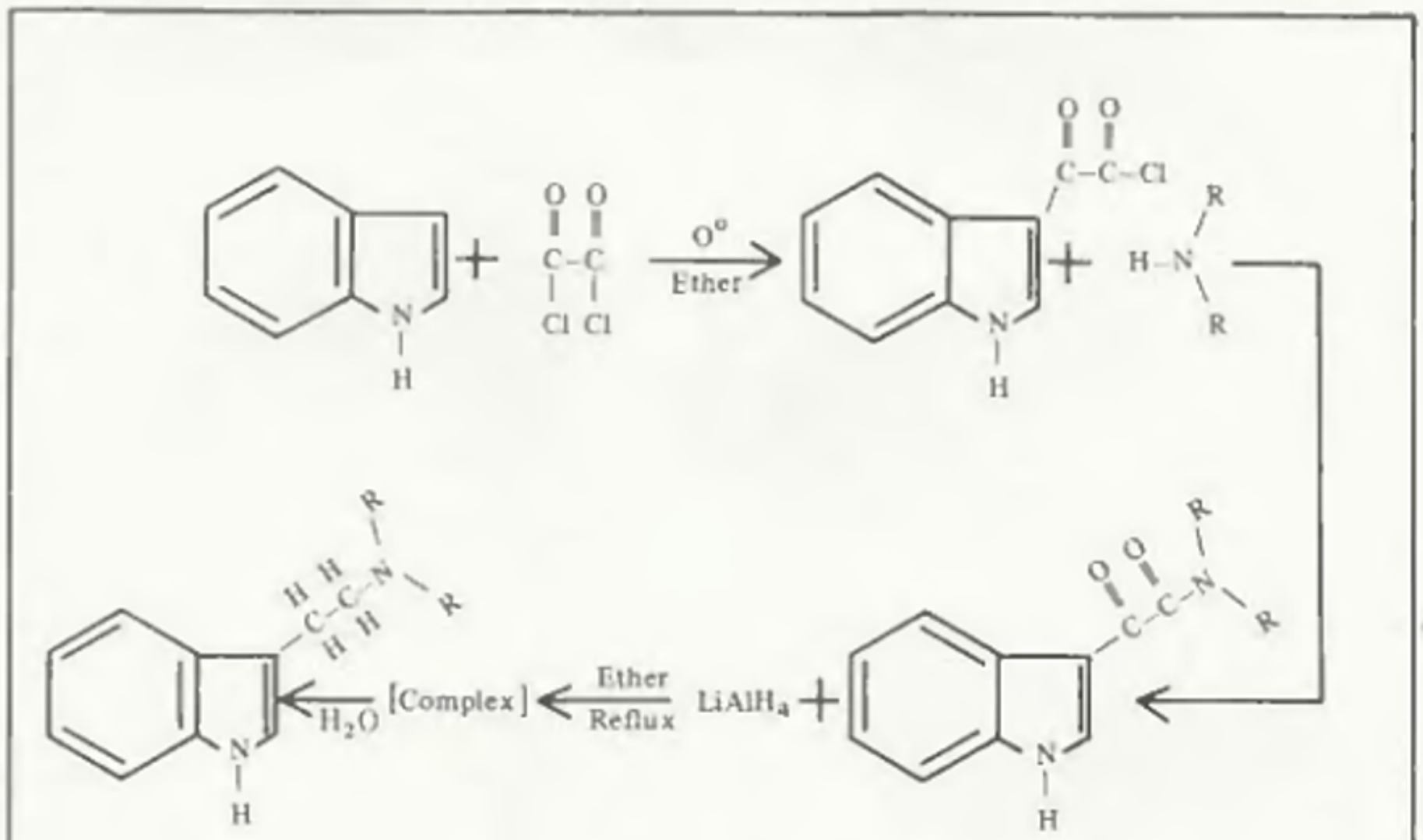
The water in the ether is used to dissolve all low-molecular weight amines.

STEP III

The crystalline amide is difficult to add to the $LiAlH_4$ mixture. A Soxhlet extractor may be used to add the amide by placing it in-between the flask and condenser. Casting it into rods or bars is one of the simplest methods. Tetrahydrofuran, if available, enables the indole glyoxal amide to be dissolved and added as a solution; a procedure which is best and fastest of all. $LiAlH_4$ is a very dangerous inflammable compound, especially so when in ether solution. The ether must be absolutely anhydrous or a violent effervescence occurs, destroying the $LiAlH_4$ and creating a fire hazard. Contact of $LiAlH_4$-ether solution with any water, damp materials, or even chemically bound water such as cellulose causes spontaneous combustion. A safety shield made from auto windshield material is a must when working with $LiAlH_4$ in any form. Handling $LiAlH_4$ is done wearing rubber gloves in a dry or inert atmosphere with a minimum of friction involved. Hydrolysis of the complex is dangerous and should be done slowly and cautiously, using an ice-bath to cool the mixture.

Difficulty in producing crystals in first time should cause no concern since many organics need seed crystals to crystallize. The syrup may be used for some purposes but be sure to save some seed crystals if you should happen to get some.

## REACTIONS




# Substituted Tryptamines

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# ERGOT CULTURE
# AND EXTRACTION OF LYSERGIC ACID DERIVATIVES

*Claviceps purpurea (Ergot)* must first be isolated as a pure culture or obtained from a maintained collection of pure culture stocks.

The culture is revitalized and prepared for innoculating a large culture by growing as a small surface culture on the medium described below for two weeks at pH 4.

| | |
|---|---|
| Sucrose | 100 g. |
| Chick pea meal | 50 g. |
| $Ca(NO_3)_2$ | 1.0 g. |
| $KH_2PO_4$ | 0.25 g. |
| $MgSO_4$ | 0.25 g. |
| KCl | 0.125 g. |
| $FeSO_4 \cdot 7H_2O$ | 8.34 mg. |
| $ZnSO_4 \cdot 7H_2O$ | 3.44 mg. |

Make up to 1 liter and adjust to pH 4 with citric acid and ammonia. Autoclave to sterilize.

Great care must be taken not to contaminate the culture, since *Claviceps* is a parasite and is taken over by any number of more vigorous strains of saprophytic fungi and bacteria.

Innoculate a number of large surface ferments in gallon jugs containing the above media, using the smaller culture by homogenizing it and using portions of it under sterile conditions.

Prior to innoculation, make an aerator (see Figure 1) by ramming a large glass tube full of cotton, fitting one hole stoppers to the ends, attaching glass tubing as shown, and attaching a stopper to fit the jugs with a vent tube to be extended to a flask containing a dilute solution of hypochlorite. Put the stoppers, tubing, and filter in a paper bag stapled shut, and autoclave it. After innoculation, carefully place the assembled aerator on the jug and force air through it into the solution.

Maintain aeration at 25° in the absence of bright lights. After ten days, adjust the culture to 1% ethanol using 95% ethanol (under sterile conditions), after which, growth is maintained under these conditions for 14 days.

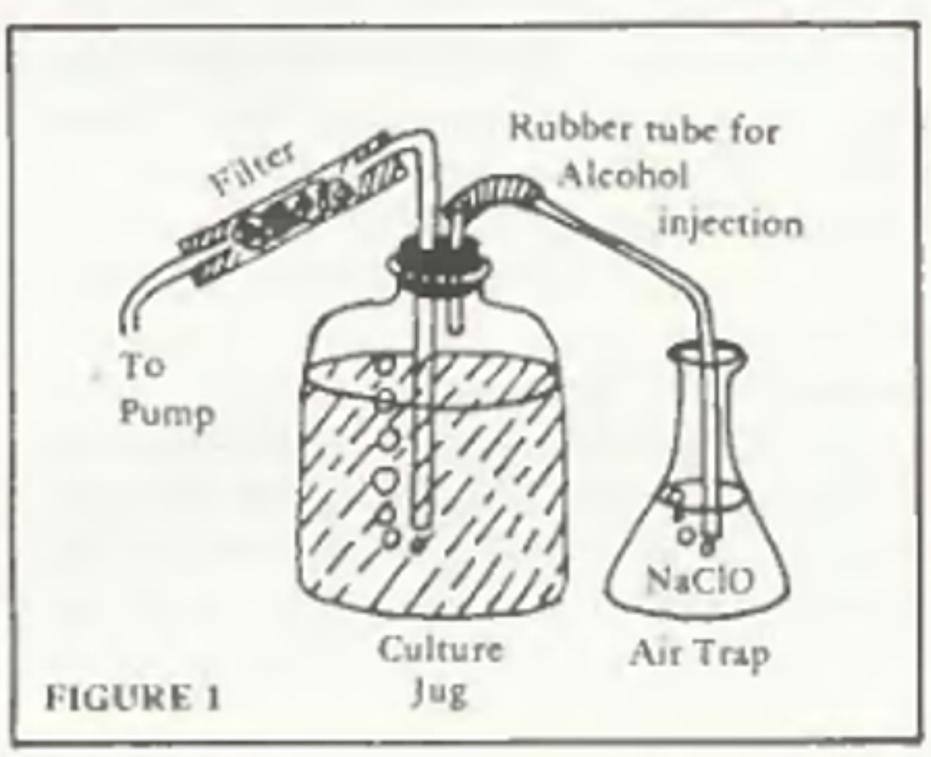

FIGURE 1

The culture is made acidic with tartaric acid and is homogenized in a blender at maturity. After an hour, $NH_4OH$ is added to adjust the pH to 9.0; and the solution is extracted with benzene or chloroform-isobutanol mixture. Extract with alcoholic tartaric acid solution and evaporate quickly in vacuo to dryness. Recover the free base as needed by making the tartrate basic with ammonia to pH 9.0 and extracting with chloroform. Evaporate the chloroform in vacuo. Protect the base from light, heat, moisture and air.

**Extraction:** Cultured ergot (see previous page), ergot sclerotia, Morning Glory seeds.

**Equipment:** Blender
Separatory funnel
Chromatography column
Flash evaporator (or hair dryer)
Long wave UV lamp

Reduce the material to a fine powder in a blender. If moist or wet, dry first, preferably in vacuo. Pack the powder in a large chromatography column as a slurry with ligroine or

lighter fluid. Soak overnight and drip (percolate) slowly until the solvent is grease-free. This takes about 5 ml/gm. of seeds, but less on ergot. When the fats are thus removed, an ammoniacal chloroform solution is washed slowly through. Prepare this solution by shaking 100 ml con $NH_4OH$ in 900 ml chloroform. The bottom chloroform layer is drawn off with the help of a separatory funnel. This chloroform wash should be dripped slowly through as soon as the ligroine fraction shows no grease film when evaporated in a watch glass. Collect and save the chloroform extract until it doesn't fluoresce on evaporation of a drop on a watch glass. Evaporate this solution using a hair drier or even better, a flash evaporator. Wash the residue with a 3% tartaric acid solution. Color the 3% tartaric solution with an acid-base indicator and estimate the number of moles of alkaloid present by titrating with this acid. Most of the residue should be dissolved or suspended. Transfer the solution to a separatory funnel, washing the evaporating vessel with extra acid. Make basic with $NaHCO_3$ solution. Add equal volume of $CHCl_3$. Shake thoroughly, let stand and remove the bottom layer. Extract again with chloroform. Reduce the combined chloroform extracts to a solid as before. Scrape the solid up with a stainless steel spatula. This powder can be used directly to make the hydrazide. Ascorbic acid is usually used as a preserving agent.

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## SYNTHESIS OF LSD-25

### Preparatory arrangements

Starting material may be any lysergic acid derivative, from *Claviceps purpurea (ergot)* on rye grain or from culture, from *Ipomea* (morning glory) seeds, or from synthetic sources. Preparation #1 uses any amide, or lysergic acid as starting material. Preparations #2 and #3 must start with lysergic acid only, prepared from the amides as follows:

10 g of any lysergic acid amide from various natural sources is dissolved in 200 ml of methanolic KOH solution and the methanol removed immediately in vacuo. The residue is treated with 200 ml of an 8% aqueous solution of KOH and the mixture heated on a steam bath for one hour. A stream of $N_2$ gas is passed through the flask during heating and the evolved $NH_3$ in the gas stream may be titrated in HCl to follow the reaction. The alkaline solution is made neutral to congo red with tartaric acid, filtered, cleaned by extracting with ether, the aqueous solution filtered and evaporated. Digest with MeOH to remove some of the colored material from the crystals of lysergic acid.

Arrange the lighting in the laboratory similarly to that of a dark room. Use photographic red and yellow safety lights since lysergic acid derivatives are decomposed by light. A weak, long wave ultraviolet source is conveniently made from the purple glass filter used in the 1950 Ford dash lighting system. A small tungsten bulb will provide enough light.

Have plenty of aluminum foil handy to cover reagents and products when light is present. Rubber gloves must be worn due to the highly poisonous nature of ergot alkaloids. A hair dryer, or, much better, a flash evaporator, is necessary to speed up steps where evaporation is necessary.

### PREPARATION #1

#### Step I – Use Yellow Light

Place one volume of powdered ergot alkaloid material in a tiny roundbottom flask and add two volumes of anhydrous hydrazine. An alternate procedure uses a sealed tube in which the reagents are heated at 112°C. The mixture is refluxed (or heated) for 30 minutes. With an open condenser, keep an inert atmosphere on the reaction. Add 1.5 volumes $H_20$ and boil 15 minutes. On cooling in the refrigerator, isolysergic acid hydrazide is crystallized .

#### Step II – Use Red Light

Chill all reagents and have ice handy. Dissolve 2.82 g of the hydrazide rapidly in 100 ml 0.1 N ice-cold HCl using an ice bath to keep the reaction vessel at 0°. 100 ml ice-cold 0.1 N $NaNO_2$ is added and after 2 to 3 minutes vigorous stirring, 130 ml more HCl is added dropwise with vigorous stirring again in an ice bath. After 5 minutes, neutralize the solution with $NaHCO_3$ saturated sol. and extract with ether. Remove the aqueous solution and try to dissolve the gummy substance in ether. Adjust the ether solution by adding 3 g diethylamine per 300 ml ether extract. Allow to stand in dark, gradually warming up to 20° over a period of 24 hours. Evaporate in vacuum and treat as indicated in the purification section for conversion of iso-lysergic amides to lysergic acid amides.

### PREPARATION #2

#### Step I – Use Yellow Light

5.36 g of d-lysergic acid are suspended in 125 ml of acetonitrile and the suspension cooled to about -20°C in a bath of acetone cooled with dry ice. To the suspension is added a cold (-20°) solution of 8.82 g of tri-

fluoroacetic anhydride in 75 ml of acetonitrile. The mixture is allowed to stand at -20° for about 1½ hours during which time the suspended material dissolves, and the d-lysergic acid is converted to the mixed anhydride of lysergic and trifluoroacetic acids. The mixed anhydride can be separated in the form of an oil by evaporating the solvent in vacuo at a temperature below about 0°, but this is not necessary. Everything must be kept anhydrous.

**Step II – Use Red Light**

The solution of mixed anhydrides in acetonitrile from Step I is added to 150 ml of a second solution of acetonitrile containing 7.6 g of diethylamine. The mixture is held in the dark at room temperature for about 2 hours. The acetonitrile is evaporated in vacuo, leaving a residue of LSD-25 plus other impurities. The residue is dissolved in 150 ml of chloroform and 20 ml of ice water. The chloroform layer is removed and the aqueous layer is extracted with several portions of chloroform. The chloroform portions are combined and in turn, washed with four 50 ml portions of ice-cold water. The chloroform solution is then dried over anhydrous $Na_2SO_4$ and evaporated in vacuo.

## PREPARATION #3

The following procedure gives good yield and is very fast with little iso-lysergic acid being produced, however, the stoichometry must be exact or yields will drop.

**Step I – Use White Light**

Sulfur trioxide is produced in an anhydrous state by carefully decomposing anhydrous ferric sulfate at approximately 480°C. Store under anhydrous conditions.

**Step II – Use White Light**

A carefully dried 22 liter RB flask fitted with an ice bath, condenser, dropping funnel and mechanical stirrer is charged with 10 to 11 liters of dimethylformamide (freshly distilled under reduced pressure). The condenser and dropping funnel are both protected against atmospheric moisture. 2 lb of sulfur trioxide (Sulfan B) are introduced dropwise, very cautiously with stirring, during 4 to 5 hours. The temperature is kept at 0-5° throughout the addition. After the addition is complete, the mixture is stirred for 1-2 hours until some separated, crystalline sulfur trioxide-dimethylformamide complex has dissolved. The reagent is transferred to an air-tight automatic pipette for convenient dispensing, and kept in the cold. Although the reagent, which is colorless, may change to yellow and red, its efficiency remains unimpaired for three to four months in cold storage. An aliquot is dissolved in water and titrated with standard NaOH to a phenolphthalein end point.

**Step III – Use Red Light**

A solution of 7.15 g of d-lysergic acid mono hydrate (25 mmol) and 1.06 g of lithium hydroxide hydrate (25 mmol) in 200ml of MeOH is prepared. The solvent is distilled on the steam bath under reduced pressure. The residue of glass-like lithium lysergate is dissolved in 400 ml of anhydrous dimethyl formamide. From this solution about 200 ml of the dimethyl formamide is distilled off at 15 mm pressure through a 12-inch helices packed column. The resulting anhydrous solution of lithium lysergate left behind is cooled to 0° and, with stirring, treated rapidly with 500 ml of $SO_3$-DMF solution (1.00 molar). The mixture is stirred in the cold for 10 minutes and then 9.14 g (125.0 mmol) of diethylamine is added. The stirring and cooling are continued for 10 minutes longer, when 400 ml of water is added to decompose the reaction complex. After mixing thoroughly, 200 ml of saturated aqueous saline solution is added. The amide product is isolated by repeated extraction with 500 ml portions of ethylene dichloride. The combined extract is dried and then concentrated to a syrup under

reduced pressure. Do not heat the syrup during concentration. The LSD may crystallize out, but the crystals and the mother liquor may be chromatographed according to the instructions on purification.

**PURIFICATION OF LSD-25**

The material obtained by any of these three preparations may contain both lysergic acid and iso-lysergic acid amides. Preparation #1 contains mostly iso-lysergic diethylamide and must be converted prior to separation. For this material, go to Step II first.

**Step I – Use Darkroom and Follow With Long Wave UV**

The material is dissolved in a three to one mixture of benzene in chloroform. Pack a chromatography column with a slurry of basic alumina in benzene so that a one-inch column is six inches long. Drain the solvent to the top of the alumina column and carefully add an aliquot of the LSD-solvent solution containing 50 ml of solvent and 1 g LSD. Run this solution through the column, following the fastest moving blue fluorescent band. After it has been collected, strip the remaining material from the column by washing with MeOH. Use the UV light sparingly during this procedure to prevent excessive damage to the compounds. Evaporate the second fraction in vacuo and set aside for Step II. The fraction containing the pure LSD is concentrated in vacuo and the syrup will crystallize slowly. This material may be converted to the tartrate by tartaric acid and the LSD tartrate conveniently crystalized. MP 190-196°

**Step II – Use Red Light**

Dissolve the residue derived from the methanol stripping of the column in a minimum amount of alcohol. Add twice that volume of 4 N alcoholic KOH solution and allow the mixture to stand at room temperature for several hours. Neutralize with dilute HCl, make slightly basic with $NH_4OH$ and extract with chloroform or ethylene dichloride as in preparations #1 or #2. Evaporate in vacuo and chromatograph as in the previous step.

**Salvage**

Neutralize all leftover solutions and residues with $NaHCO_3$ and evaporate in vacuo to low volume. Extract with ammoniacal chloroform and evaporate the extract to dryness. This residue may be run through the whole process again and more LSD will be produced.

**Storage and Use**

Lysergic acid compounds (among them LSD) are unstable to heat, light and oxygen. In any form it helps to add ascorbic acid as an anti-oxidant, keeping the container tightly closed, light-tight with aluminum foil, and in a refrigerator.

Packaging for use presents many possibilities, partially due to the incredibly small dosage involved. First a bio-assay of the purified solution is made, then it may be measured by the volume of the solvent it is in. The solvent may be evaporated onto a weighed, calculated amount of some inactive powder such as chalk, sugar or baking soda. This bulky powder may be easily encapsulated in weighable portions. It is advantageous to add a trace of dry ascorbic acid to the dried powders. Sugar cubes offer a handy but extremely notorious method of dispensing. Other methods are without number, here being offered just a few occasionally used by the criminal element. Gelatin capsules are coated with the liquid solution and the capsules filled with an inert substance. Decoys such as this inert mixture might include a trace of brown color, a trace of quinine for fluorescence, and a trace of some relatively non-toxic compound which nearly mimics the infra-red spectrum of LSD. For transport, a smuggler might evaporate a considerable amount onto a pocket handkerchief or onto a sheet of paper, providing the solution was properly decolorized before such treatment. These underhanded methods are used by criminals to avoid puni-

tive action by law enforcement enthusiasts.

One gram of pure LSD, if used in a truly enlightened careful manner can be the door to a magnificent experience to nearly 3,000 individuals. Used furtively and in ignorance, the same amount may bring terrible confusion and abject terror to nearly one thousand of these.


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## STP

In the future many more hallucinogenic drugs will be developed, some of which will be safer and more pleasant than any available now; others treacherously poisonous or otherwise unreliable as the drug known as STP often is.

STP, the Dow Chemical Company drug known to them as DOM, is an amphetamine closely related to benzedrine. The correct name is 2, 5 dimethoxy 4 methyl amphetamine or 1,(2, 5 dimethoxy 4 methyl) phenyl 2 amino propane. This drug is synthesized in the same manner as benzedrine, provided the difficult to obtain intermediate, 2, 5 dimethoxy 4 methyl phenyl acetone, is available. Any attempt to order this compound will bring the purchaser to the attention of agents of the Justice Department. To synthesize this compound involves lengthy and expensive procedures and a suitable synthesis is not available to the editors. Many other methoxy amphetamines are active hallucinogens similar to STP, but most have the disadvantage of having too long an effect, which can be quite unpleasant if one has a bad trip.

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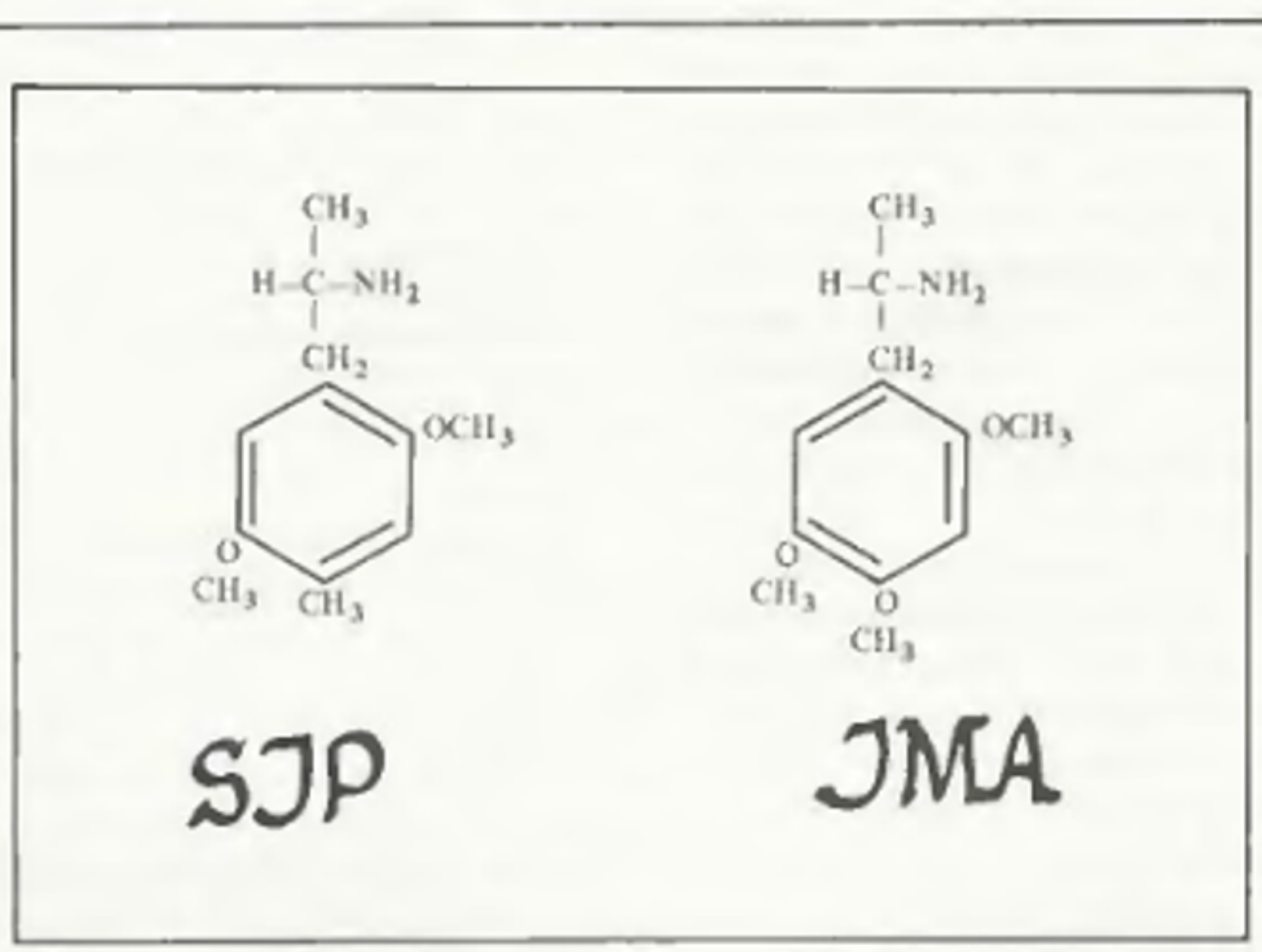


### Procurement of Necessary Items and Prohibitions Thereon

The apparatus necessary for these operations can easily be obtained from scientific supply houses or in some instances, can be made at home. The laboratory where the work is to be done must not be in the U.S. unless it is with special FDA or Bureau of Narcotics and Dangerous Drugs approval. Criminals and fugitives from the law use either a legitimate commercial or educational laboratory; or construct a home lab with adequate water, ventilation, a fire extinguisher and tight enough security so neither friends nor neighbors are aware of its existance.

Merely the attempt to purchase one of certain chemicals necessary in several of the procedures will bring an immediate investigation of the buyer by the Bureau of Narcotics and Dangerous Drugs. Failure to provide a good (and we do mean legal) use for any and all of these chemicals will mean that the victim not only does not get the reagents, but will be watched by the Bureau's secret agents for several years. Legitimate labs are liable to inspection and may be required to keep an accounting of restricted chemicals. Law-abiding organizations are appalled by the chemist who obtains reagents from his friendly underground supplier or from a friend who works for a large laboratory or supply house. Beware of the small laboratory supply company; they are often a police front although they catch more innocent lab operators than drug pushers.

Illegal drug makers use longer, more involved synthetic methods because the commoner starting reagents make detection by the Bureau more difficult. These same methods are included for the legitimate chemist only because the starting materials are less expensive and commonly obtainable. Keep in mind, however, anyone purchasing a number of chemicals (especially organics) who is not connected with a legitimate lab will likely draw suspicion.

Illicit extractions, such as mescaline from Peyote, are detected when watchful agents notice amateurs buying a sequence of simple chemicals and solvents. To any trained person such an assortment of materials is plainly saying, "I am extracting mescaline." The outlaw chemist therefore is quite cautious in purchasing certain groups of materials from single companies or from several companies at one time.

Any legitimate chemist must not attempt to purchase any of the following in the U.S., unless he has a solid and legitimate use for them.

**Restricted or watched reagents**
**(descending order of importance)**

Lysergic acid
Ergotamine (and other ergot alkaloids)
Olivetol
Citral
3, 4, 5 Trimethoxy benzaldehyde
Lithium aluminum hydride
Boron trifluoride
3, 4, 5 Trimethoxy benzoic acid
Oxalyl chloride
Diethylamine
Dimethylamine
Sulfur trioxide (Sulfan B)
Trifluoroacetic acid
Hydrazine (anhydrous)
Indole
Pulegone
3, 5 Dinitrobenzoyl chloride
2, 6 Dinitro toluene

It must be clearly pointed out that many things throughout this manual have recently become unlawful and no one should ever break the law. Therefore, anyone attempting to pursue the course outlined herein (in countries where prohibited) must be willing to shoulder the burden of persecution which may fall upon him. Since the religious user and chemist are unfortunately not immune to legal retribution, the editors sincerely hope that no one will be forced to martyr himself through his incorrect use of this guide-book.

### Wobble Stirrer

Use an induction motor (brush motors cause sparks) and rig a pulley system to decrease the speed to 200-400 rpm. Attach an eccentric-plate to the drive shaft and make a long stirrer by putting a steel welding rod in a polyethylene ¼ inch tube, making a flat paddle at the bottom (see Figure 4). The stopper must have an hour glass shaped hole or flexible diaphram which will allow the rod to wobble, but not necessarily turn. Always remember that some hot solvents (chloroform or benzene) cause polyethylene to crack or dissolve and substitutes may have to be found in certain cases. (Teflon)

Weighted polyethylene balls or small marbles may be used as stirring aids.

### Supports

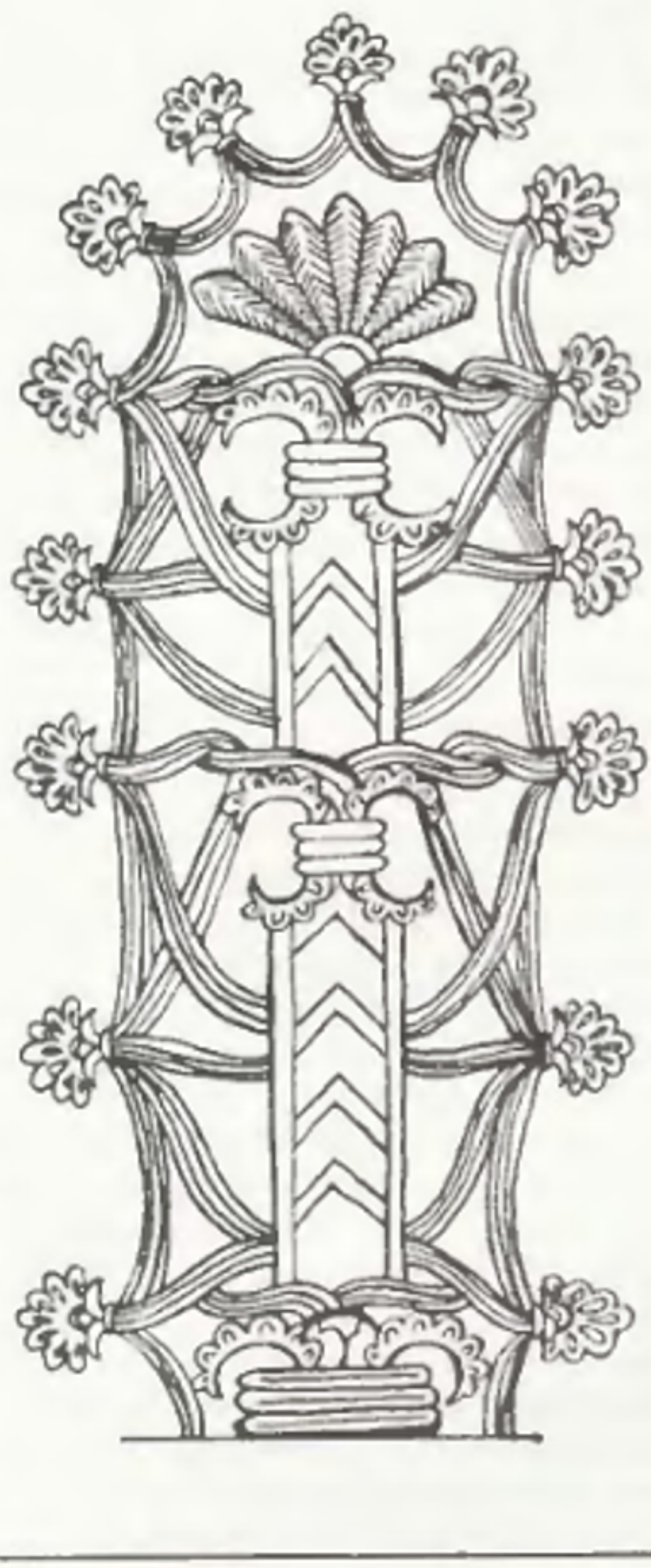
Ring stands and rings may be made out of steel plate and rod. (see Figure 5)

### Vacuum Pump

A very good and inexpensive vacuum pump may be made from the compressor of an old refrigerator or freezer. Certain refrigerator compressors have three leads. The center lead is the starting winding and is necessary only to overcome the inertia of the compressor during the first two or three seconds of operation. Connect the leads to two switches using a single pole-single throw switch and a momentary SPST switch (see Figure 6). Operate both switches together, releasing the momentary switch as soon as the compressor motor starts. A cold trap (see Figure 6) made with a thermos bottle full of dry ice and a solvent such as acetone or gasoline through which a U-tube passes will help protect the pump from the corrosive gasses encountered in many vacuum distillations. A primary trap of ice water will help keep the colder trap from plugging quite as fast as it is sometimes prone to do.

### Drying Oven and Evaporator

Use a kitchen stove oven, turning the setting to 200° F and propping the door 2" open to prevent overheating. For inflammables, use only an electric oven, preferably one without glowing elements. For removing large volumes of volatile solvent, use an old hair dryer in a well-ventilated area.



## HOMEBUILT LABORATORY EQUIPMENT

Lab equipment is one of the major costs in setting up a good home lab. With a little ingenuity, most necessary equipment and a few instruments may be built with little cost and only a moderate amount of time and effort. One must judge for oneself if certain equipment may be built or if it must be purchased. Where glassware is needed, for instance, it is often most economical in the long-run to buy apparatus with ground glass joints since many reactions need all-glass equipment and tight joints to contain corrosive and dangerous chemicals.

Many pieces of equipment are overpriced due to small demand or willingness to spend excess grant moneys to equally willing suppliers on other equipment. On the following pages are instructions and diagrams giving most of the information necessary to the building of several different pieces of laboratory apparatus. For further ideas, consult the "Amateur Scientist" section in the Scientific American magazine.

### Heating Mantles

Heating mantles cost about $30-$40 from scientific houses but can be made at home to fit any use for about $1 each.

Use asbestos insulating cement available from a heating contractor's supply house or make your own from a mixture of 1 lb. portland cement to 1 lb. asbestos fiber. Prepare the flask or beaker to be used as a form by painting a layer of hot paraffin on the surface to be covered. (see Figure 1) Mount the model upside-down and begin by pressing the doughy-wet cement in a layer. Arrange coil nichrome heating element in the desired pattern and cover with more cement. Read directions on element spool for measuring enough wire to give the desired wattage. Mold a good layer of the insulating cement over the element to give good insulation. Anchor the ends of the elements to heavier terminals for easy connection to a voltage controller. Dry the mantle in air or in an oven before connecting any electricity, as electrolysis will dissolve the element.

### Magnetic Stirrer

Get a small induction motor as is used in small fans and tape-recorders. Use a horseshoe magnet with a hole in the curve or make a clamp base. Do not try to machine alnico. Secure and line-up the magnet on the motor shaft (see Figure 2) and mount the motor in a suitable framework. An asbestos shingle works as a heat-proof cover and will not drag on the magnet as a metal will do. Fashion the stirbar by sealing a small bar magnet in glass or polyethylene or buy a teflon stir bar. The speed is controlled with the use of a variac voltage regulator.

### Balance

The balance may be built almost entirely out of wood, heavy wire, glass tubing (split lengthwise) and single-edge stainless ejector-type razor blades.

The critical factors in setting up a balance are:

1. Distance between side-arm knife edges to the central knife edge must be exactly the same.
2. The center of gravity must be slightly below the central knife edge fulcrum.
3. Sensitivity is adjusted to optimum by weighting the indicator needle and by bending the arms.

See Figure 3 for the approximate layout and construction.

Common weights:

dime - 2.5 grams
nickle - 5.0 gms
quarter - 6.125 gms
half - 12.25 gms.

FIGURE 1

## heating mantles

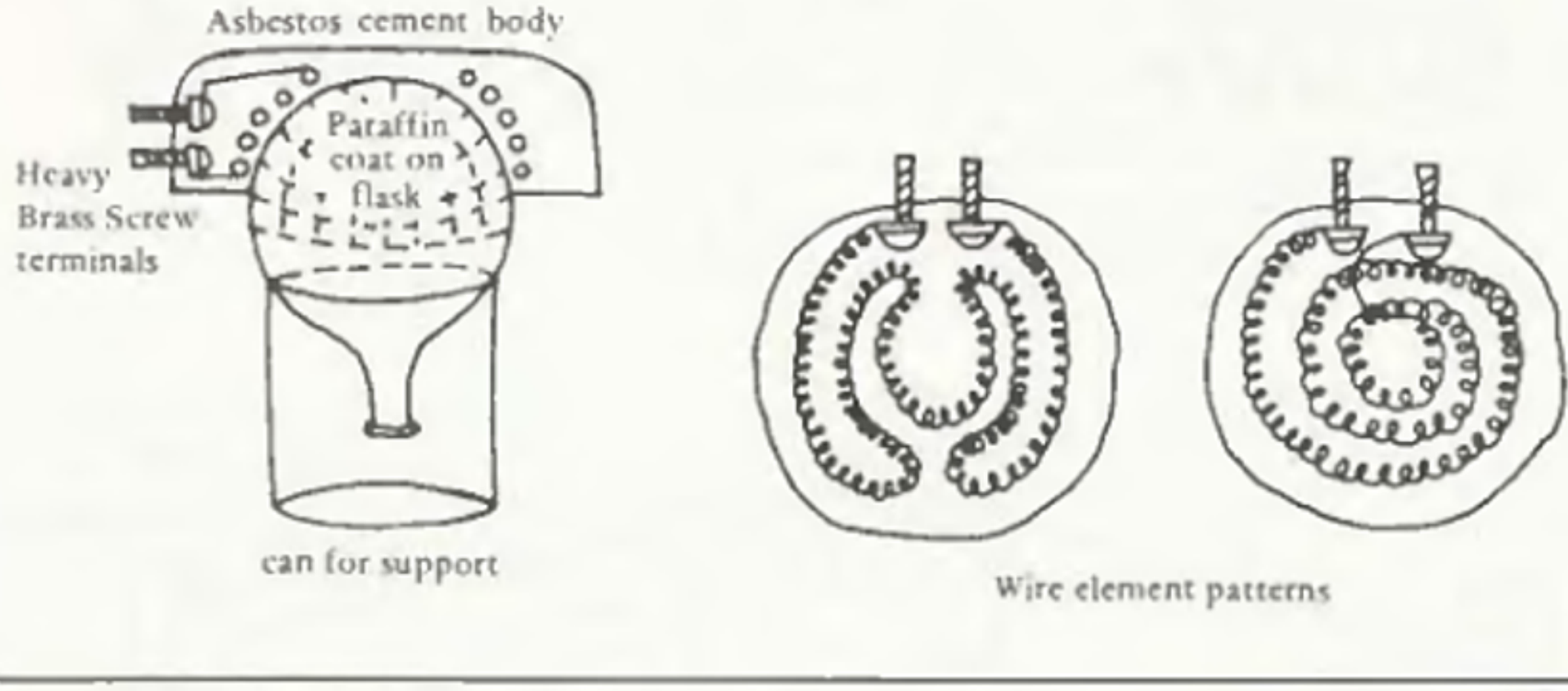



FIGURE 2

## magnetic stirrer

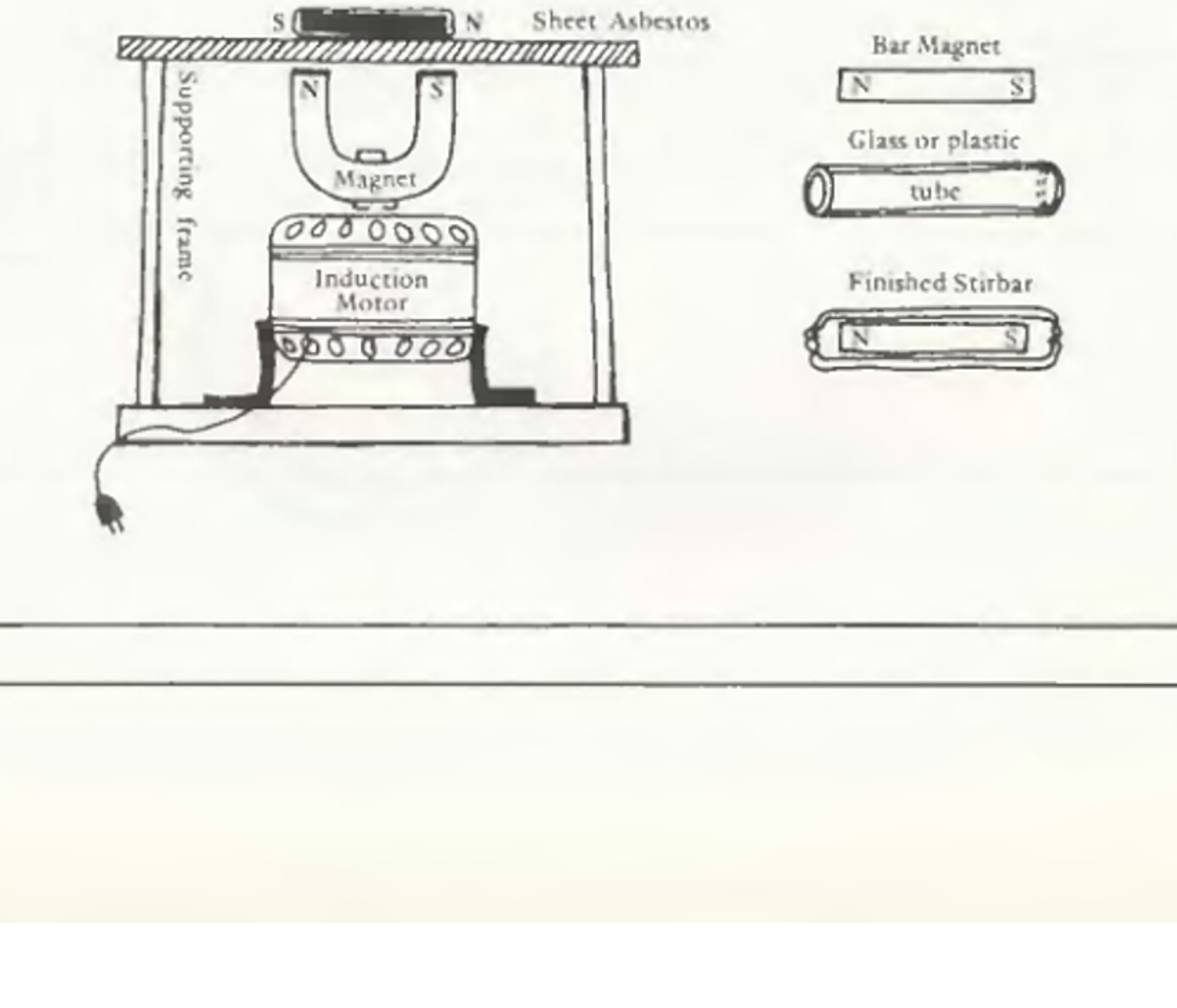


FIGURE 3

# balance

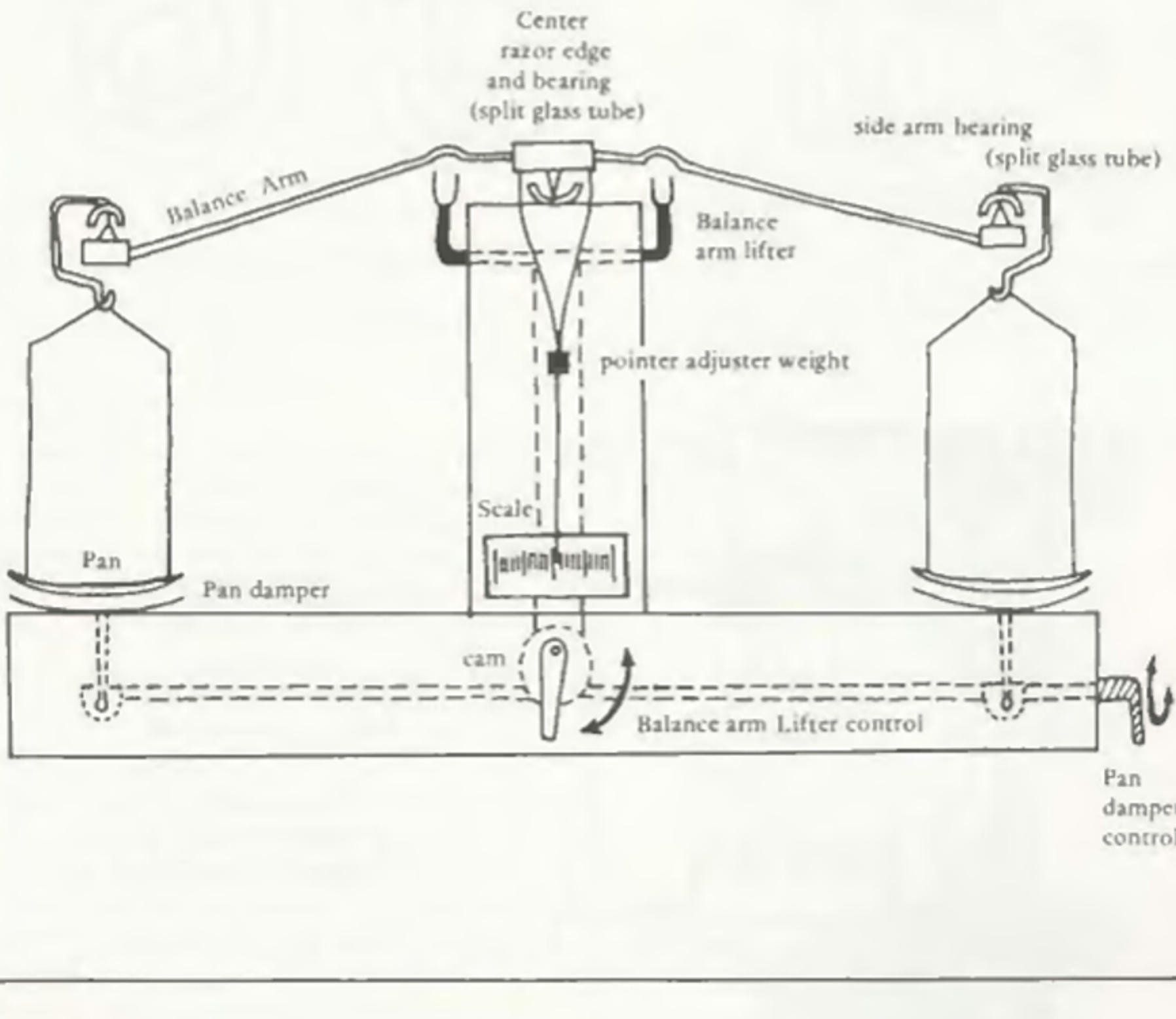


FIGURE 4

# wobble stirrer

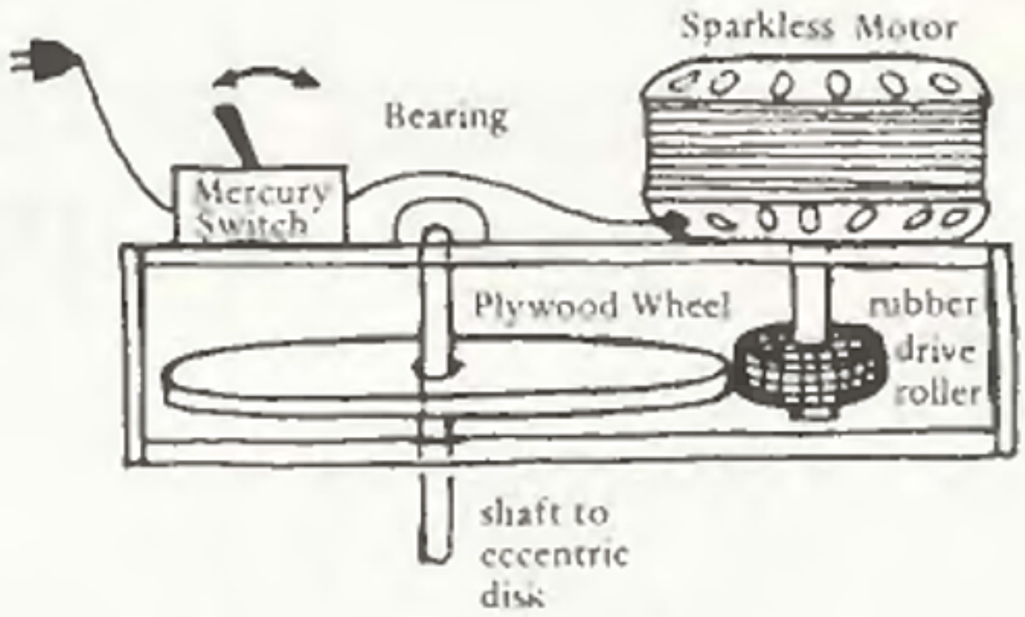


FIGURE 5

## supports

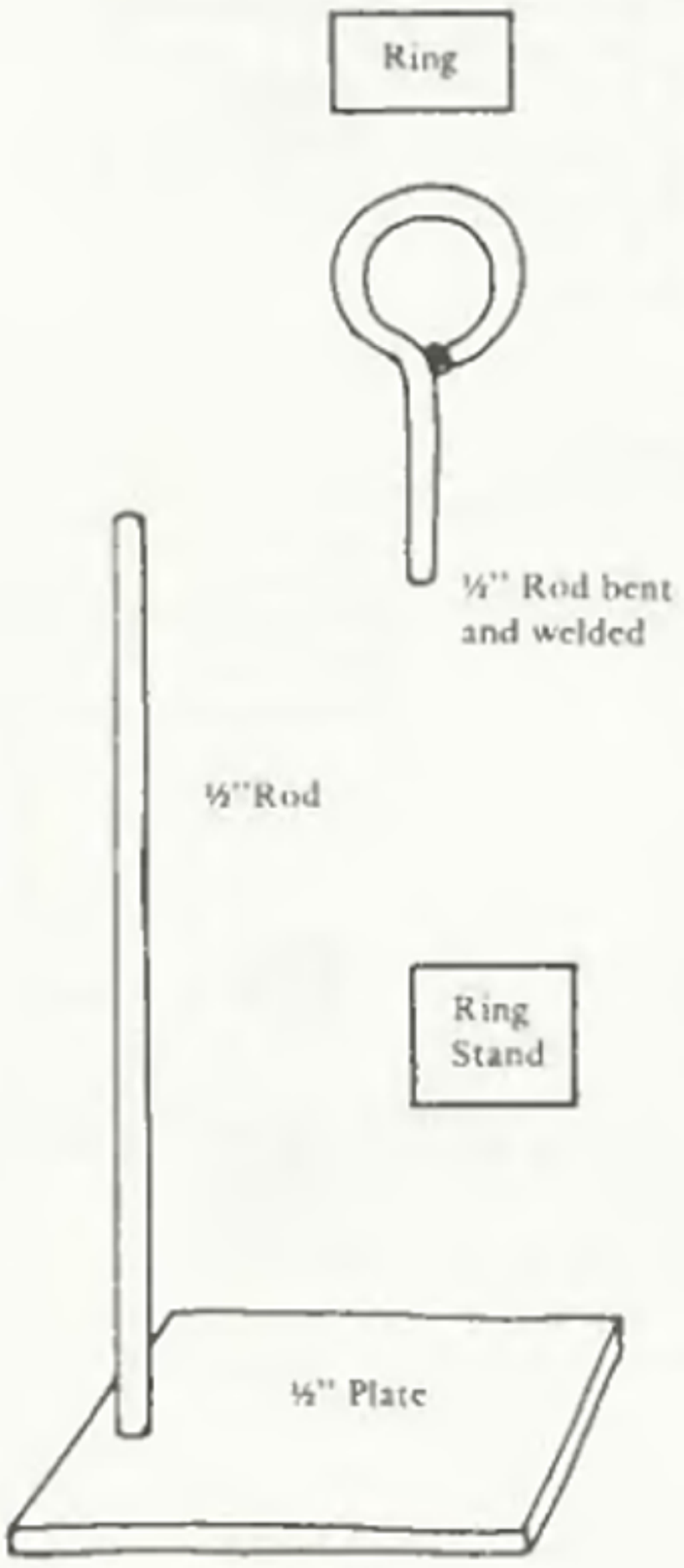
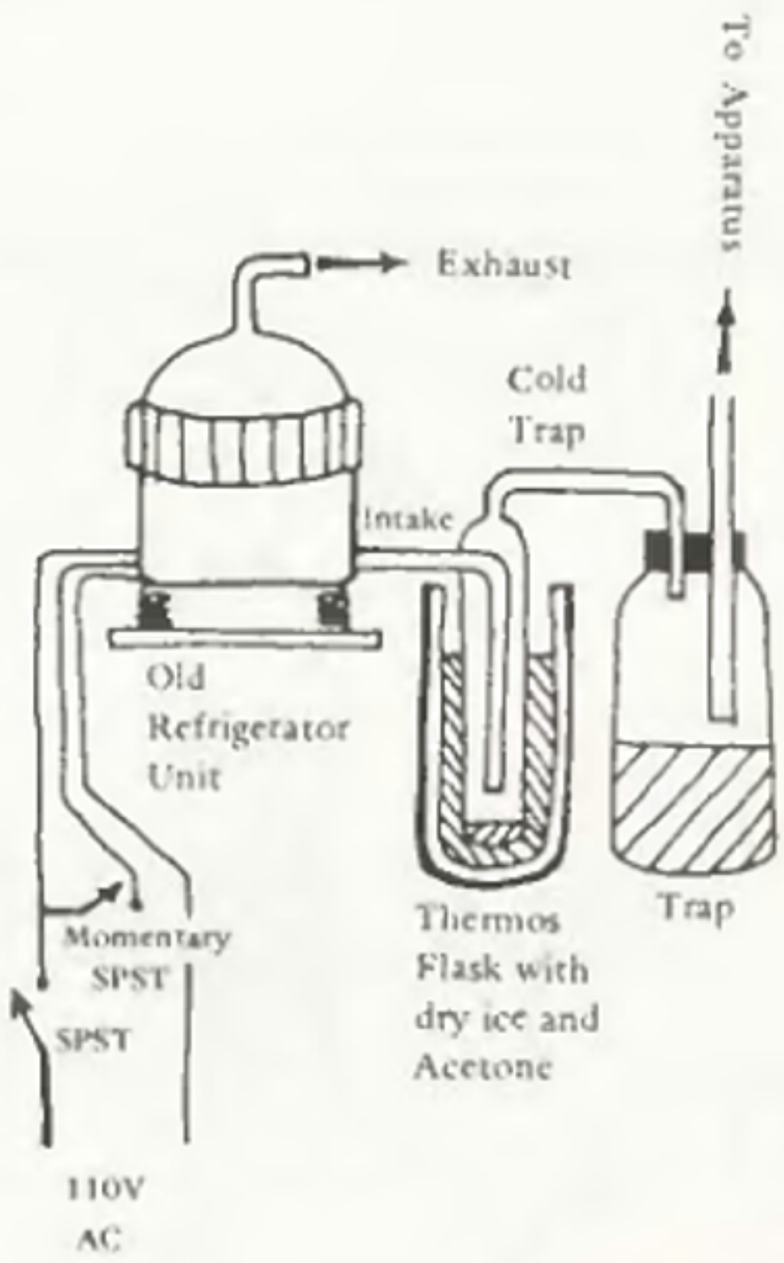



FIGURE 6

## vacuum pump



The use of psychedelic drugs in an attempt to seek personally relevant symbols is as ancient as the compulsion to symbolize. The illustrations in this book are intended to indicate the structural similarities of symbols from many cultures and periods, derived from both drug and non-drug inspired sources. Diverse religious and meditional approaches to the central themes of creation, death, and recyclation within a universal consciousness indicate a preoccupation with personal awareness and harmony which is also the present impetus for the use of psychedelic drugs.

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half-title page – Chinese talisman against evil influences. The yin-yang is surrounded by the eight trigrams; the sequence determines the intent.

title page – the Lotus and the egg – The Hindu/Buddhist representation of the relationship between the Cosmic egg and the Buddha; between visual-outer and inner knowledge. This resembles in form the Assyrian tree on page 50 and the Buddhist wheel on page 34. The path leads through receptivity to unity, a concept also of the Christian chalice.

p. 1 "Om mani padhma hum"

p. 3 Buddist "parm" in form of Lotus seed

p. 4 Charlestown gravestone - prerevolution colonial

p. 6 - Medieval sign for "the intellect in action."

p. 7 - *Shah Abbas* - Persian ornament, found in 17th century rug, representing the lily; fertility and happiness. Named after brilliant 16th c. ruler of Iran.

p. 9 - glyph from Monte Alban, stella 9; god descends from the sky.

p. 10 - *Allah is the Past and the Future* - Moslem talisman.

p. 11 - from stone *camposanto* or gravemarker, Tecolote, Mexico.

p. 26 - the *Shou* - Chinese symbol based on the spiral, meaning happiness.

p. 29 - carved mushroom stone - Formative (Miraflores, Kaminal juyu) Guatamala.

p. 31 - triple spiral - from Syrian cylinder seal, 18-17th c. B.C. The spiral is a multicultural motif representing eternal motion. The closed spiral, as in the infinity symbol, is especially powerful. Three was a very important number in early Mesopotamia, meaning generative union – male, female, child.

p. 34 - Buddhist Wheel of the Law (Darmachakra) Sanchi, India. This is a common image in Judaism & Christianity also – the axle tree ascends from earth to sun, through the sun door, into the world beyond.

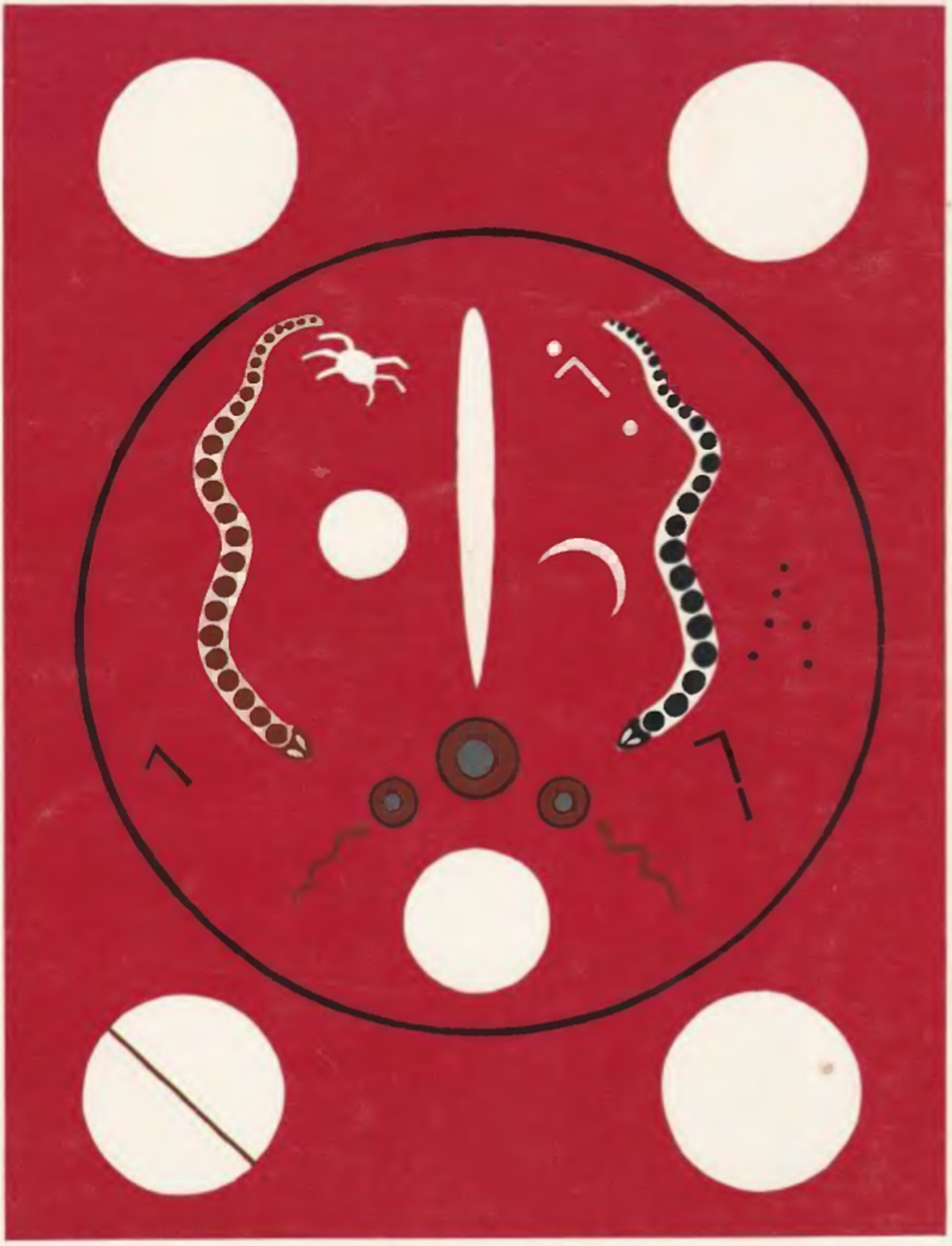
p. 42 - the all-seeing eye, common to all major religions as a symbol of omnipotence.

p. 46 - the god of Light, Ahuramazda - Achsemenian cylinder seal about 500 B.C.

p. 50 - tree-of-life from an Assyrian bas relief.

✦

back page – female mandrake - Medieval woodcut representing the mandrake plant which was used by the ancients for it's magical aphrodesiac properties probably chosen for it's shape, rather than drug content.

*Back cover:* "The Coming of Age Initiation": Diegueño Indian Sand Painting, Mesa Grande, San Diego County, California. Near the Spring equinox, this sand painting functioned as the focal point for puberty rituals of the young people of the mesa. The ritual was originally accompanied by use of a vision-producing weed in a drink, but this practice was sometimes fatal to the initiate and was therefore abandoned.

Designed by Eje Wray